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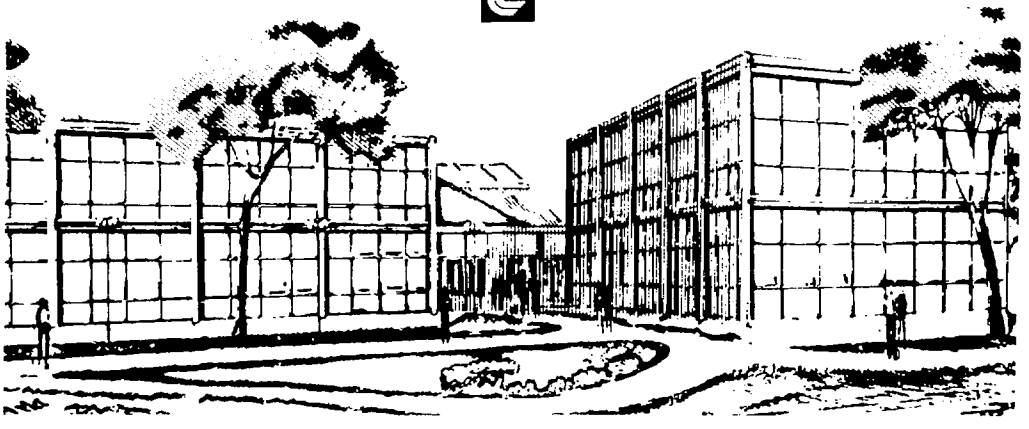

RAPID AUTOMATED NUCLEAR CHEMISTRY*

A Review of Rapid Chemical Separations, Techniques, and Application for the Isolation and Study of Short-Lived Fission Products†

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1. INTRODUCTION

The utility of information derived from the study of fission products ranges from the quest for the understanding of our origins to problems associated with the design and monitoring of today's energy sources. Certainly, input into cosmological problems of the R-process requires accurate base information derived from fission-product data on masses, half-lives, and nuclear structure (Schramm and Norman 1976; Takahashi 1977; Beck 1977; Clayton 1968). The design of reactor cooling systems requires not only the measurement of decay heat by integral methods (Dickens et al. 1978) but also, for more advanced designs, summation calculations based on information concerning the decay properties of individual contributors (Reich 1977; Prussin et al. 1974). The investigation of fission yields by spectroscopy techniques requires fission-product decay metrology (Denschlag 1977). While pursuing the acquisition of this fission-product metrology, we simultaneously are acquiring a base of information that allows us to explore fundamental properties of nuclei under conditions of high excitation energy and large neutron excess. In turn, some of the longer-range applied problems can be solved only by obtaining answers to fundamental questions such as the nature of beta-delayed neutron emitters, including their energy spectra, daughter-level population ratios, and the related question of beta strength (Kratz 1978, 1979; Reich 1977).

Because the fission process produces such an overwhelming number of nuclides, detection equipment cannot cope simultaneously with all the many β rays, γ rays, and neutrons emitted after the fission process. Therefore, we must find ways to isolate the individual fission-product species in order to study their properties in detail. With no universal technique available to snatch a bundle of single species out of the chaos of fission, two methods of isolation have emerged:

A-Separators

Z-Separators

Most A-separators (ISOL systems, etc.) provide some sort of continuous source of selected mass (see, for example, the talks at this workshop by Carrey, Fogelberg, Schrader, Sistemich, and Wollnik). However, these separators provide simultaneously several or all atomic numbers of a given fission-product mass-yield chain. Selection of individual isotopes generally is brought about by various techniques of timing (moving tape collectors, etc.) as well as preselection of certain elements such as the noble gases (TRISTAN-I) or alkali metals (OSTIS). The alternate method for isolation of fission products uses Z-separation. For the worker who has too long focused on only physical methods, I include Equation 1.1.

$$\text{Z-Separation} \equiv \text{Chemistry} \tag{1.1}$$

In regard to the study of fission products one might legitimately ask, what is chemistry? If we are to include all techniques that identify or isolate by atomic number then we must include techniques such as the multiparameter work of the type done by Wilhelmy (1969) and Cheifetz and Wilhelmy (1974). For our purposes we should limit our consideration to those that use chemical effects to isolate the fission product. Currently this does not include the art of ion sorcery (sic Sorcery) (Fogelberg 1979, Wollnik 1979). We shall discover later that the understanding and design of ion-source operation is very much akin to one subcategory of nuclear chemistry being explored (see, for example, Zendel 1978; Zendel et al. 1978). I would highly recommend that an important future direction for nuclear chemists is the investigation into the understanding and development of ion-source chemistry so that we may have ion sourcery rather than ion sorcery.

As seen in Figure 1.1 the majority of the elements produced, for example in the thermal fission of ^{235}U, extend from an atomic number of approximately 30 to slightly greater than 60. Suggestion of a certain priority for elements that can be isolated more effectively by chemistry is shown in Figure 1.1, where the fission-product distribution for thermal fission of uranium is compared to the output of ISOL systems such as TRISTAN and OSTIS (Wohn 1977; Chrien 1977). The nuclei we want to explore require that we isolate isotopes that represent only a small fraction of the entire amount and after they have decayed away leave large amounts of residual long-lived, unwanted activity. Thus, for fission products the technique we find most attractive is the use of fast radiochemical separations in an automated fashion to produce sources for nuclear measurements. Rapid Automated Nuclear Chemistry (RANC) can be thought of as the:

Z-separation of
Neutron-rich
Isotopes by
Automated
Methods. (Meyer 1979 Note 1)

The range of RANC studies of fission and its products is large. In a sense, the studies can be categorized into various energy ranges from the highest where the fission process and particle emission are considered, to low energies where nuclear dynamics are being explored. Table 1.1 gives a list, by no means exhaustive, of examples of current research using RANC on fission and fission products. The remainder of this text is divided into three parts. The first contains a discussion of the chemical methods available for the fission product elements, the second describes the major techniques, and in the last section, examples of recent results are discussed as illustrations of the use of RANC.

TABLE 1.1. Examples of Current Research Using Rapid Automated Nuclear Chemistry of Fission

Area of Investigation	Principal Investigator and Institution	Area Reference
Fission process	H. O. Denschlag (Mainz) A. C. Wahl (Washington U. St. Louis) K. Wolfsberg/ B. Erdal (LASL)	Denschlag 1977 Wahl 1977
Particle emission		
Beta decay and neutron emission	K.-L. Kratz (Mainz) S. G. Prussin (Berkeley)	Kratz 1979
Beta decay processes	S. G. Prussin (Berkeley) K.-L. Kratz (Mainz)	Klapdor and Wene 1979
Half-lives far from stability (Stellar R-Process consequences)	G. Herrmann (Mainz/GSI Darmstadt) N. Trautmann (Mainz)	Schramm and Norman 1976 Takahashi 1977 Hillebrandt and Takahashi 1976
Nuclear properties		
High spin γ-ray traps	H. C. Griffin (U. Michigan)	
Shell model at high isospin	E. A. Henry (LLL)	Lane 1979
New regions of deformation	SISAK collaboration at Mainz	Sheline 1976
Multiparticle effects	W. B. Walters (U. Maryland)	Alaga, Paar, and Sips 1975
Shape coexistence	R. A. Meyer (LLL)	Ragnarsson, Nilsson, and Sheline 1978
Collective interactions		
Bosons		Iachello 1978
Quadrupole/octupole interaction		Sheline 1976

2. CHEMICAL ISOLATION OF ELEMENTS AVAILABLE FROM FISSION

Even before fission was ushered into world knowledge (Hahn and Strassmann 1939) and some of its products first identified by chemistry (Hahn and Strassmann 1940), several techniques for studying radioactivity were already in use. The experiments that identified 55-s ^{220}Rn used evaporation (Owens 1899); the first gas jet, albeit slow, was used by Rutherford (1900) to identify the nature of "emanation" observed in his earlier experiments (Rutherford 1899; Chadwick 1962). It was the chemist Soddy who "blurted out, 'Rutherford, this is transmutation'" and went on with Rutherford to formulate the theory of spontaneous radioactive decay (Trenn 1977). Even subsecond half-lives were studied by recoil methods and rotating disks that were adapted from spark studies (Schuster and Hemsalech 1899) to identify activities with periods as short as 2.0 ms (Moseley and Fajan 1911; Meyer 1979 Note 2).

The study of fission and its products gave rise to the development of numerous radiochemical techniques (see, for example, Stevenson and Hicks 1953; Miskel 1950; Finston and Miskel 1955). Glendenin (1949) reported values for the independent yields of ^{133}I and ^{134}I, as did Pappas for antimony and tellurium (Pappas 1951, 1953). Further developments can be illustrated by the series of investigations by Wahl and his students at Washington University. They developed chemical techniques for 60-s Ag (Wahl and Bonner 1952), 120-s I (Wahl 1955), ~4-s Kr and Xe (Wahl 1958), 30-s Sr (Wahl et al. 1962), 25-s La (Troutner, Wahl, and Ferguson 1964), 50-s Sn (Wahl and Nethaway 1963), 45-s Sb (Troutner, Wahl, and Ferguson 1964), 20-s Y (Norris and Wahl 1956), 6-s Rb (Wahl, Norris, and Ferguson 1966), and 5-s Sn (Erdal, Wahl, and Ferguson 1971) in order to study fission-product chemical yields.

Modern rapid radiochemical research owes much to the work of Prof. Gunther Herrmann and the Institut für Kernchemie of Johannes Gutenberg Universität Mainz. Early work in the field has been presented (Herrmann 1966)

and given an in-depth review (Herrmann and Denschlag 1969). More recent work centered at Mainz has been reviewed in published form (Trautmann and Herrmann 1976) and brought up to date in unpublished form (Trautmann 1978).

Examples of recent work on the chemical isolation of elements from fission are given in Table 2.1. These range from separations for Ga ($Z = 31$) to those for the rare earths ($Z > 58$). Where possible, examples of batchwise and continuous separations are cited. These two techniques are discussed in Section 3. The usual tendency, when one discusses chemical separation, is to order the homologues in chemical groups. Because we are addressing the use of these chemical separations to isolate elements from fission, the following discussion of recent chemical-separation work is centered around groupings corresponding to their position with respect to increasing atomic number and the fission-mass yield curve. Again, for a comprehensive review of these procedures the reader is referred to the works of Herrmann and Trautmann.

2.1 Elements Lighter Than Gallium ($Z < 30$)

Little work is reported for this region. The use of dimethylglyoxine for the isolation of Ni from fission products has been investigated (Sekine and Boba 1978). Some further demand for development of separation techniques may arise with the availability of fissioning systems that emphasize the lower atomic number elements (Griffin 1979 PC).

2.2 Low Z Shoulder, Light Mass Peak ($_{31}Ga$, $_{32}Ge$, $_{33}As$, $_{34}Se$)

The separation of these elements depends predominately on the use of volatilization. Several new isotopes of Ge have been identified by separations based on the formation of volatile $GeCl_4$ via passing Cl_2 gas through HCl solution of irradiated uranium (del Marmol and Fettweis 1972; del Marmol and van Tegchelt 1972). The possibility of rapid purification of Ge with use of purpurogalline (Tchavirian and Bevillard 1951) has been

TABLE 2.1. Recent Rapid Radiochemical Investigations

Z	Element	Technique (First author of representative article and year)	
		Autobatch	Continuous
31	Ga		Solvent extraction (Liljenzen 1972)
32	Ge	Volatile chloride/direct exchange (del Marmol 1972/Larson 1976)	
33	As	Volatilization (Kratz 1973)	
34	Se	Volatilization (Kratz 1970)	Thermal chromatography (Zendel 1978)
35	Br	Se volatilization/Br extraction (Hicks 1979/Slaughter 1976)	Solvent extraction (Skarnemark 1979)
36	Kr	Examination (Xe Trapping) (Wahl and Daniels 1952/ Herrmann 1969)	
37	Rb		
38	Sr	Precipitation (Herzog 1974)	
39	Y	Solvent extraction (Rengan 1976/Klein 1976)	Solvent extraction (Aronsson 1978)
40	Zr	Solvent extraction (Trautmann 1972)	Gas phase (Zvara 1970, 1971)
41	Nb	Sorption on glass (Ahrens 1976)	Gas phase (Zvara 1970, 1971)
42	Mo	Solvent extraction (Tittel 1977)	Thermochromagraphy (Bayer 1978/Niedhardt 1972)
43	Tc	Solvent extraction (Trautmann 1976)	Gas phase (Matschoss 1979) Solvent extraction (Skarnemark 1979)

TABLE 2.1 (Continued)

Z	Element	Technique (First author of representative article and year) Autobatch	Continuous
44	Ru	Volatile oxide (Fettweiss 1975)	Gas phase (Matschoss 1979)
45	Rh		Gas phase (Matschoss 1979)
46	Pd		Solvent extraction (Aronsson 1970)
47	Ag	Exchange with solids (Bruchle 1976)	
48	Cd		(Grapengiesser 1973)
49	In	Exchange (Erdal 1971)	Solvent extraction (Wahl 1979)
50	Sn	Volatilization (Naeumann 1972)	
51	Sb	Volatilization (sorption) (Rudolph 1977/Lane 1979)	Thermochromatography (Trautmann 1976)
52	Te	Volatilization (extraction) (Folger 1969/Hicks 1978, 1979)	Thermochromatography (Zendel et al. 1978)
53	I	Exchange with solids (Schussler 1969, 1972)	Gas phase (Zendel 1978)
54	Xe	Examination (see Herrmann 1969)	(Isotope separations) (Henry 1972)
55	Cs	Electrophoresis (Tamai 1972)	
56	Ba	Exchange (Yaffe 1979/Herrmann 1969)	
57	La		Solvent extraction (Björnstad 1978)
58	Ce	Ion exchange (Klein 1975)	Solvent extraction (Aronsson 1974)

TABLE 2.1 (Continued)

Z	Element	Technique (First author of representative article and year)	
		Autobatch	Continuous
59	Pr		Solvent extraction (Skarnemark 1976)
60	Rare Earths	High-pressure chromatography (Schädel 1977/Gschneider 1979, 1980)	Solvent extraction (Skarnemark 1979; Gehrke 1978).

investigated (Larson 1976). The use of volatile hydride production for the separation of As and Se has been used extensively (Kratz and Herrmann 1970, 1973; Henry et al. 1979).

Continuous separations have been performed for Se and Ga. Selenium has been separated using gas-phase chemistry (see Zendel 1978 for an excellent investigation of the parameters and techniques of gas-phase chemical techniques). An early use of the centrifuge technique was for the separation and identification of short-lived Ga isotopes (Liljenzen 1972).

2.3 Low Z Light Mass Peak (35Br, 36Kr, 37Rb)

Here the main recent chemical work has been on the isolation of bromine from fission. Presumably, the lack of activity in radiochemical separation results from the availability of Kr and Rb in high abundances from on-line isotope separators (see, for example, references in Bailleul et al. 1975; Klapish 1976; and Henry 1972). Some work on fast continuous separations for Rb has been performed to measure fission yields (Aumann and Weismann 1978). For bromine, direct extraction is difficult on a short time-scale; several attempts at isolation of short-lived Br activities have given poor results (Morita and Prussin 1978) while similar techniques using batchwise solvent extraction have proved useful (Nuh et al. 1972). However, gas-phase separation has allowed the isolation of ^{88}Br to ^{92}Br (Kratz, Lauppe, and Herrmann 1975). High-purity ^{86}Br and ^{87}Br in ratios of ^{86}Br to ^{87}Br from 25 to 1, to as much as 1 to 1.5 have been obtained for spectroscopic studies by extraction of bromine from previously separated selenium via the hydride production method (Hicks 1978).

2.4 High Z of Low Mass Peak (38Sr, 39Y, 40Zr)

Chemical isolation of 24-s ^{95}Sr by an oxalate precipitation from fission products has been performed by batchwise separation (Herzog and Grimm 1974). Solvent extraction using a solvent-pair system containing HCl and

HDEHP has been applied to the isolation of yttrium activities from fission products (Rengan and Griffin 1976; Trautmann 1978) while short half-lives of both Y and Zr have also been investigated with use of ion exchange resins (Klein 1975; Trautmann 1978).

2.5 High Z Shoulder, Low Mass Peak (41Nb, 42Mo, 43Tc, 44Ru and 45Rh)

The batchwise separation of Nb, Mo, Tc, and Ru isotopes from fission products has been reviewed (Trautmann et al. 1972; Trautmann 1978). The predominant technique for separation of these elements is the use of various extractants in a solvent extraction-based procedure. A typical procedure is found in the description of the isolation of short-lived Mo isotopes (Tittle et al. 1977) or Ru isotopes (Franz and Herrmann 1978). An alternate procedure based on the oxidation of Ru to form RuO_4 by means of Cl_2 gas has been used to study isotopes down to 1.5 s in half-life (Fettweis and del Marmol 1975). The 80-s isotope ^{109}Rh has been studied by batchwise separations (Franz 1978).

Several continuous procedures have been developed for the isolation of these elements. An example of continuous Tc chemical separation will be used later to illustrate the Mainz-based SISAK-consortium continuous chemistry capability (Trautmann 1978, 1979; Skarnemark 1979). Bächmann and coworkers (Bächmann et al. 1976; Matschoss and Bächmann 1979) have investigated the separation of Tc and Ru on a continuous basis from a spontaneously fissioning ^{252}Cf source. They used both H_2O and O_2, separately and in combination, while varying the gas-flow rate, temperature of transfer tube, and temperature of an adsorption trap (Matschoss and Bächmann 1979). Quite different transport properties are found using the two transport gases at various temperatures and partial pressures. A thermochromatographic column on Ag-coated quartz powder (Eichler and Domanov 1974) was used to rid of iodine. The selective separation of Ru required the removal of Tc and I by a

collection trap using volatilization with HCl at 150°C. The Tc was studied by the same technique with the exception that HCl was introduced after adsorption of I on a Ag-trap followed by trapping of the Ru and accumulation of Tc on a room-temperature cold finger (Matschloss and Bächmann 1979). It is interesting to note that these techniques are being extended to the measurement of thermodynamic data (Heinlein and Bächmann 1979) and that gas-phase techniques based on earlier work (Neidhardt et al. 1972; Weber et al. 1973) are being used to investigate the neutron-deficient Mo and Tc isotopes (Bayar et al. 1978).

2.6 Valley Elements (46Pd, 47Ag, 48Cd, 49In)

Little work has been published on the separation of these elements. The study of short-lived Ag isolated by exchange has been investigated (Brüchle 1976; Trautmann et al. 1976; Trautmann 1978). The identification of ^{116}Pd was made isolating Pd with solvent extraction techniques and using the rapid-phase separation device SISAK (Aronsson, Ehn, and Rydberg 1970). Separation of indium from fission products using SISAK techniques based on earlier solvent extraction work (Erdal 1966) is being explored (Wahl 1979).

2.7 Low Z Shoulder, Heavy Mass Peak (50Sn, 51Sb, 52Te)

Like their lighter mass homologues these three elements share the technique of hydride batchwise and gas-phase continuous chemistry as their predominant mode of isolation. The technique of hydride production has been described in several reviews (Herrmann and Denschlag 1969; Trautmann 1978; Trautmann and Herrmann 1976, 1978) and will be used later in this text as an example of the rapid autobatch technique. The continuous separation but discontinuous source measurement of tellurium is described by Zendel (Zendel 1978; Zendel et al. 1978).

2.8 Low Z High Mass Peak (53I, 54Xe, 55Cs, 56Ba)

A number of investigators have used continuous techniques to isolate these elements. Gas-phase techniques using volatile methyl halides (Kratz and Herrmann 1970) have been used to isolate short-lived iodine isotopes (Kratz et al. 1975). An interesting application for rapid isolation of iodine on Ag-coated quartz powder deserves further attention (Matschoss and Bächmann 1979; Eichler and Domanov 1974). The investigation into the oxidation state of fission-fragment iodine should prove useful in better design of chemical fission-yield experiments (Walters 1978). The pseudochemistry of a gas-recoil jet system has been used to isolate short-lived cesium for yield measurements (Aumann and Weismann 1978). Investigation of the isolation of cesium by electrophoresis also has been explored (Tamai et al. 1972). There is a dearth of reported work on the isolation of barium. Some initial work using exchange techniques (Yaffe and Stone 1979) is in progress.

2.9 High Z Heavy Mass Peak (57La, 58Ce, Z > 58)

Numerous solvent extraction procedures have been used to isolate the short-lived La and Ce nuclei. These mostly use the SISAK continuous solvent extraction technique such as the isolation of ^{150}Ce (Aronsson et al. 1974), ^{143}La (Björnstad et al. 1978), $^{144-146}$La (Skarnemark et al. 1978). The isolation of Pr isotopes by the milking of SISAK-separated La has been used to study short-lived Pr isotopes (Skarnemark et al. 1976).

The fast separation of the rare-earth nuclei are more on the time scale of a few minutes and currently depend on the use of high-pressure liquid chromatography (Sisson, Mode, and Campbell 1972; Mode and Sisson 1979). A recent review of these techniques can be found in Radiochemia Acta (Schädel, Trautmann, and Herrmann 1977, 1979). Some development work on the use of centrifugal contactors (Leonard et al. 1978) using a ^{252}Cf source is in progress (Baker et al. 1978). For a comprehensive presentation of rare-earth

properties and chemistry techniques, the reader is referred to the recently published four-volume work of Gschneider and Eyring (1979).

2.10 Heavier Homologues of Fission Products

It should be pointed out that a close companion area of chemistry is the effort to develop isolation techniques for heavy elements (HE) and superheavy elements (SHE). These elements are fission homologues, so developments in rapid chemistry techniques for them can be applied to the isolation of homologue fission products and vice versa (Müller and Lindner 1976; Brüchle 1976; Hulet et al. 1976; Bächmann et al. 1976; Chudinov et al. 1976).

3. TECHNIQUES USED IN ELEMENTAL ISOLATION

3.1 Types of Systems

The techniques for chemical separation of fission products can be broken into two types: batchwise separation and continuous initial separation. However, one must recognize at the outset that many "continuous" techniques in fact produce the final measurable in incremental (batchwise) amount, and because of effects such as source holdup, times are not necessarily instantaneous. The decision as to whether to employ batchwise or continuous conditions often depends upon the problem to be attacked and the source of fission products available (viz., a high-flux reactor, a pulse TRIGA-type reactor, a 14-MeV neutron generator, etc.). However, when compared, the efficiency of batchwise operation relative to continuous system generally shows that activities with half-lives down to a few seconds can be studied by batchwise operation. This has been shown by Stevenson (1977), who assumed:

1. The detector used is to be operated up to, but not above, a counting rate A_0.

2. The "continuous system" is to be operated in such a fashion that the detector operates constantly at a maximum rate A_0.

3. The "batch process system" will deliver samples periodically to the detector; at the time of delivery, the detector rate will be A_0 and this rate will fall exponentially with the half-life of the desired species. (N.B. No allowance is made for the artificial apparent lengthening of the half-life by dead-time effects.)

4. The batch-system detector remains on for a duration of k_1 half-lives; it is then turned off for a duration of k_2 half-lives while the latest sample is discarded and a new one brought into position.

5. Both systems are to be operated in such a way that the abundance of the desired species is optimized relative to the interfering species.

During a period of $(k_1 + k_2)t_{1/2}$ the number of events N_c recorded by the continuous system is

$$N_c = (k_1 + k_2)t_{1/2}A \tag{3.1}$$

and the number recorded by the batch system is

$$N_b = A_0 \int_0^{k_1 t_{1/2}} \exp(-\lambda t)\ dt = \frac{A_0}{\lambda}\left[1 - \exp(-k_1 \lambda t_{1/2})\right] \tag{3.2}$$

or

$$N_b = \frac{A_0 t_{1/2}}{\ell n 2}\left[1 - \exp(-\lambda k_1 t_{1/2})\right] = \frac{A_0 T_{1/2}}{\ell n 2}\left[1 - \exp(-k_1 \ell n 2)\right] \ , \tag{3.3}$$

so that efficiency η is given by

$$\eta \frac{N_b}{N_c} = \frac{1 - \exp(-\lambda t_1 t_{1/2})}{(\ell n 2)\left[k_1 + k_2\right]} \frac{1 - \exp(-k_1 \ell n 2)}{(\ell n 2)\left[k_1 + k_2\right]} \quad . \tag{3.4}$$

Since k_2 may be determined by the chemical and mechanical constraints of the system it is desirable to maximize η for a fixed k_2, i.e.,

$$\frac{\partial \eta}{\partial (k_1)} = 0 = \frac{(\ell n 2)\ \exp(-k_1 \ell n 2)}{(\ell n 2)(k_1 + k_2)} - \frac{1 - \exp(-k_1 \ell n 2)}{\ell n 2 (k_1 + k_2)^2} \ , \tag{3.5}$$

so that

$$k_2 = \frac{\exp(k_1 \ell n 2) - 1}{\ell n 2} - k_1 \tag{3.6}$$

determines the counting time k_1 (for a given sample-change time k_2) to give maximum efficiency. That efficiency is

$$\eta \max(k_2) = \frac{1 - \exp(-k_1 \ell n 2)}{\dfrac{1 - \exp(-k_1 \ell n 2)}{\exp(-k_1 \ell n 2)}} = \exp(-k_1 \ell n 2) \quad . \tag{3.7}$$

Stevenson then plots $k_1(k_2)$ and $\eta \max(k_2)$ as a function of k_2, although because of the nonlinear nature of Equation (3.6) and the simple form of Equation (3.7) it is in practice much easier to evaluate both k_2 and η max for various values of k_1. We can also plot $k_1 + k_2$, since because of the finite rabbit-speed there is a constrained lower limit on cycle time.

These curves are shown in Figures 3.1.1 and 3.1.2. With these he estimates the efficiency of the batch process, given the half-life of the desired species and the mechanical constraints of the system.

Example: Assume a 2-second half-life

a 2-second sample-changing time

a 6-second limit on the cycle time.

Then $k_2 = 1.0$, $\eta = 0.37$ and $k_1 = 1.4$, therefore $k_1 + k_2 = 2.4$ but $k_1 + k_2 > 3.0$ so that the sample changing time is not the limiting factor.

If we set the cycle time $k_1 + k_2 = C$ (in half-lives) then

$$\eta = \frac{1 - \exp(-k_1 \ell n 2)}{C \ell n 2} \tag{3.8}$$

and

$$\frac{\partial \eta}{\partial (k_1)} = \frac{\exp(-k_1 \ell n 2)}{C} , \tag{3.9}$$

which is everywhere positive.

Since in the example the sample-change time is not the limiting factor, maximum efficiency can be gained by counting as long as possible and using only the minimum time to change the sample. Equation (3.5) now gives $\eta = 0.36$ for $k_1 = 2.0$, $k_2 = 1.0$.

Example: Assume, instead, a 3-second half-life so that $k_2 = 0.67$,

$k_1 + k_2 \geq 2.0$ then η max = 0.44, $k_1 = 1.2$ and

$k_1 + k_2 = 1.87$; the cycle time is still limiting;

Equation (3.5) gives $\eta = 0.43$.

Example: Assume a 4-second half-life: $k_2 = 0.5$, η max = 0.48 and $k_1 = 1.05$, $k_1 + k_2 = 1.55$. The cycle limit is $k_1 + k_2 \geq 1.50$, and we therefore are limited by the sample-change time to an efficiency of ~1/2.

Example: Now assume only a 1-second half-life; we know $k_1 + k_2 = 6$, $k_2 = 2$ and, therefore, $k_1 = 4$. Equation (3.5) gives $\eta = 0.23$.

It is seen by this analysis, assuming the chemistry can be performed within the given time limits, that the batchwise relative efficiency is acceptable even for a 1-second half-life.

One more limitation must, of course, be considered; the time interval between the irradiation of the sample and the arrival of the purified sample at the counter must be adjusted to take account of the optimization of the desired species with respect to interfering radiations.

3.2 Continuous Systems

Perhaps the ultimate goal of performing continuous chemistry is to mate a continuous chemical isolation system to the output of an on-line isotope separator, thereby attaining unique separation by first mass and then by atomic number (Rudstam 1979). The first step in this direction is on-line or continuous chemical separation. One must recognize, of course, that in part we are already doing the reverse of this through chemical selectivity of certain types of ion source design. Perhaps the earliest continuous isolation of radioactivity was the separations of Rutherford (1900). For liquid-phase separations a breakthrough has been the ability to perform rapid-phase separations with small-volume high-speed centrifuges (Rydberg 1969, 1979; Reinhardt and Reinhardt 1967, 1969; Andersson et al. 1969). These centrifuges have been used to perform on-line (continuous) separation of fission products from liquid delivery systems (Aronsson 1974) or mated to gas-jet systems

(Trautmann et al. 1975). More recently, investigators have been turning toward the use of pure gas-phase chemistry for the isolation and study of fission products (see, for example, Zendel 1978; Zendel et al. 1978; Bächmann et al. 1976, 1978; Aumann and Weismann 1978; D'Auria 1977). Although these systems are described in the literature it is appropriate here to review some aspects of their operation.

A schematic drawing of a SISAK experiment is shown in Figure 3.2.1. Removal of the fission products from the fissioning site is performed by either liquid flow through a target chamber with $UO_2(SO_4)_2^{-2}$ complex absorbed on an anion exchanger (Aronsson 1974) or by use of a gas-jet recoil system (Trautmann et al. 1975; Silva et al. 1977). The latter designs use clusters in a carrier gas and are based on concepts discussed elsewhere (see, for example, Macfarlane and McHarris 1974; Dautet et al. 1973; Wollnik et al. 1975; Wilhelm et al. 1974). The gas jet is sent to a degassing unit and then on to the first stage of a SISAK unit as diagrammed in Figure 3.2.2.

The rapid-phase separation that occurs at the H-10 centrifuge can be understood by following the flow through the internal parts of the centrifuge as diagrammed in Figure 3.2.3. (Reinhardt 1979; Rydberg 1979). The mixed phases enter through a side port (A) after being thoroughly mixed in a Kenix mixer, and arrive at the inlet chamber (B) where they are accelerated to the rotational speed of the inner bowl. Upon acceleration the mixed phases are forced into a separation chamber (C) containing several isolated chambers symmetrically arranged about the central shaft. The separation chamber baffles (D) are slanted such that the heavier phase, forced outward by the centrifugal force, is also forced downward toward the heavy-phase collecting chamber (E). Here it encounters a stationary spiral wheel (F) that, in concert with the rotational speed of the liquid, acts as a pump and forces the liquid up and out the heavy-phase outlet (G). Conversely, the light-phase path

is through the upper chamber (H) to a light-phase pump (I), then up and out a light-phase exit (J). A more detailed description will appear in the published literature soon (Rydberg et al. 1979). Performance features are discussed in detail by Aronsson (1974). As diagrammed in Figure 3.2.1, the centrifuges are run in series with mixers to perform initial extraction (Figure 3.2.1/II), selective extraction (III), and final back extraction cleanup (IV). The final centrifuge is used to perform cleanup of the extractant before it is recycled. Experiments can, of course, use from one (e.g., Griffin, Skarnemark, and Meyer, unpublished) to several mixer-centrifuge stages (e.g., Skarnemark 1977).

An example of this use is the current set of experiments by the SISAK-Mainz-Oslo-Göteborg collaboration to isolate the short-lived Tc isotopes as diagrammed in Figure 3.2.4 (Skarnemark 1979; Trautmann 1978). At the first centrifuge (C1) the extraction yields are Tc (as TcO_4^-) ~100%, Zr and Nb ~5%, while all other elements are not detected in the organic phase. At the second centrifuge (C2) the Tc, Zr, and Nb are back extracted and Tc measured at the aqueous phase output of the third centrifuge (C3). The configuration shown is for measuring the shorter half-lives of Tc (^{107}Tc and ^{108}Tc); for longer-lived ^{106}Tc (36 s), NaOH is added after C3 so that solution was approximately 0.1 M HNO_3. Then the solution is passed through an anion exchange column with a holdup time of one to two minutes. With this setup, three-parameter coincidence ($\gamma\gamma t$) and angular correlation experiments have been possible and attempts are now being made (Skarnemark 1979) to measure the decay energy (masses) of the Tc isotopes 106, 107, and 108 via β-γ coincidence techniques. The use of this technique perhaps could be enhanced by applying an electrostatic spray of design similar to Figure 3.2.5 (Meyer 1959). This would allow the continuous deposition of a thin source suitable for electron or neutron energy spectra measurements.

A totally gas-phase approach has been demonstrated by the Mainz group (Zendel 1978; Zendel et al. 1978; Trautmann 1978). In this technique a volatile species such as Te reacts with ethylene (Sidgwick 1950). As shown in Figure 3.2.6 an ethylene-N_2 jet is passed over a uranium target and then passed through a series of traps to remove contaminants and homologues. The gas enters a reaction chamber where it is heated. The tellurium is completely absorbed in a charcoal trap, while contaminated noble gases pass by. As an alternative, the Se trap can be replaced by a collector for the study of Se activities (Zendel 1978). These techniques also have been used to isolate activities of Nb, Mo, Tc, Sn, and Sb (Zendel 1978). Perhaps an interesting combination for a very rapid isolation of iodine activities would be the use of a moving tape made of silver granules deposited on Mylar*. This could possibly be used for rapid removal of very short-lived iodine (see, for example, the iodine trap used by Matschoss and Bächmann 1979).

The ideal continuous system should deliver a single isotope. One way to accomplish this is to first separate by mass using an ISOL system, then perform continuous chemistry. Such an advanced system has been developed at Studsvik by Rudstam and coworkers (Rudstam 1979; Grapengiesser 1972, 1973). This system uses first the chemical selectivity of an ion source, then the mass separation capability of OSIRIS and, finally, gas-phase thermal chromatography. In the last stage the ion beam for OSIRIS is brought through a pair of slits to a heating element. The ions then diffuse down a quartz column placed at a right angle to the ion beam. This quartz column has a

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*Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.

thermal gradient such that the undesired elements are condensed on the walls and the desired element is collected at a collection site.

3.3 Autobatch Techniques

The alternate technique to continuous separation is to perform chemical isolation of discrete amounts of gross fission products. This batchwise separation can be repeated a number of times until sufficient data are accumulated. Because of several factors, including level of radioactivity handled, reproducibility of chemical separation, etc., these operations generally are automated. Herrmann and Denschlag (1969) described the early batch-separator system at Mainz that grew into their "building block" setup and spawned many significant studies. Any attempts at description of rapid chemistry must include (at least by tradition!) a sketch of the classic Mainz design as shown in Figure 3.3.1, which is a modified version of that shown in Tittle et al. (1977) on the identification of $^{103-107}$Mo. Here, a breakable rabbit (A) of fissioning solution is smashed at a collection site (B) and chemicals added (C). Subsequent reagents are added (D) to allow purification of the desired species, which is finally collected on a movable filter (E) that is shot to a counting station (F).

The "smash and shoot" design of isolation and delivery has been efficient for studies at a pulsed reactor, as witnessed by the large number of publications that have borne the Hallmark of Mainz over the years. As shown in Section 3.1, batchwise separation can be competitive with continuous separation of approximately one-second half-lives if the recycle time is held to a minimum. For example, Trautmann (1978) described an experiment on short-lived antimony performed by a discontinuous method that required 4 weeks to obtain 210 runs but was collected in a single week of continuous experiments. If the recycle time is reduced to even 30 seconds, the same experiment could be accomplished in 24 hours. With the potential of automated

batchwise separation (autobatch) in mind, systems using slightly different techniques have been developed.

An illustration of a fully-automated batchwise separation system is the one in use at the Nuclear Chemistry Division at Lawrence Livermore Laboratory. The LLL autobatch system had its origins in the designs operated by Prof. Charles Coryell's group at MIT (Landrum et al. 1972; Walters 1971) and is similar to concepts used at the University of Michigan (see Griffin 1979). Our original system (Landrum 1972) was designed to study isotopes of Group VA elements (Figure 3.3.2) with approximately 100-s half-lives. The full cycle of operation for our automated system is diagrammed in Figure 3.3.2A. Rabbits containing doubly encapsulated 1-mg samples of U-235 in solution are shot into the reactor from an automatic rabbit launcher. A computer controls the loading and firing from a magazine that holds 38 rabbits. A microprocessor loads and sends the rabbit through a pneumatic tube (activated by nitrogen gas) to the core of the Livermore Pool-Type Reactor (LPTR). The sample is irradiated for a specific time, then the microprocessor sends it through a second pneumatic tube to a receiver located in a chemistry laboratory hood approximately 45 meters from the core of the LPTR. Once emplaced in the laboratory catcher, the rabbit is transferred to a second position and punctured by a double concentric needle that extracts the fission product, adding acid in the process.

Purging with N_2 gas (Figure 3.3.3) cleans out the gases produced during fission and precludes their daughter activities from being observed in subsequent spectroscopy measurements. The actual chemical separation of Sb from the other fission products is made upon addition of sodium borohydride ($NaBH_4$) to the acidified fission products and carrier. The stibene and arsine (gaseous SbH_3 and AsH_3) are transported through a $CaSO_4$ trap that eliminates other hydrides produced by $NaBH_4$ reaction. The final step is

destruction of the stibene with KOH in ethanol; the homologue AsH_3 passes through. This final step leaves the Sb on a substrate in front of a counting station. As soon as the final chemical step is finished, the microprocessor turns on the spectrometers for measurement. An alternate procedure uses a $CaSO_4$/NaOH trap for the study of short-lived As isotopes, as illustrated in Figure 3.3.4.

An important feature of the microprocessor control is the capability for simultaneous operations; for example, spectrometers are cleaned from the chemistry system and new reagents replenished during the measurement period (Figure 3.3.2). This reduces the cycle time and, hence, increases the number of measurements that can be made in an operational day. The actual number of measurements, then, depends only on the length of the measurement segment, which can vary from a short time (if gamma-gamma-time three-parameter coincidence measurements are being made on a short-lived activity such as 10-s Sb-134 decay) to 30 minutes in our experiments to measure the independent-fission-yield isomer ratio of $^{132}Sb^{g}$ vs $^{132}Sb^{m}$. The measurement rate has been greater than 1000 isolation/measurement cycles in one 12-hour LPTR operating day. The improvements to our system are shown in Figure 3.3.5. A feature that saves a few tenths of a second but significantly decreases the amount of "down time" during an experiment has been the installation of an impaling needle rather than the catcher, transfer-block, needle-puncture sequence which we used in our earlier system. A similar system is under construction and testing at LASL (Erdal and Fowler 1979).

The operation of highly automated systems offers many advantages. For example, it is possible for a single experimenter to operate them. However, experimental realities force a minimum staff to be present to monitor chemical performance, change traps, and maintain adequate reservoir levels and miscellaneous supplies. Features such as limited capacity of the autoloader

(38-rabbit capacity) serve useful purposes. For example, a photocell that detects when the autoloader has only 5 rabbits remaining is useful as it is attached to an alarm for the changing of traps. (Typically a single $CaSO_4$/NaOH trap will service 25 single separations.) The monitoring of chemical purity generally is performed by attention to the individual spectra (each experiment is stored separately on computer disc when doing a multiscaling operation). However, this can be further automated by monitoring areas of the spectra, having the computer scan selected portions of each spectrum (Stevenson 1979).

Design features can also dictate the type of chemical reactions that are used for main-line separations. For example, the LLL-autobatch uses the borohydride reaction exclusively to produce the Group VA and VIA elements whereas others have used predominantly the zinc reaction (see, for example, Trautmann et al. 1976). Although the borohydride reaction is not as efficient as the Zn reaction for the production of some elements it lends itself to a higher degree of automation. As shown in the illustration of an early reaction vessel (Figure 3.3.6), the borohydride can be introduced in solution by metered aliquots and the old fission products washed out after the hydride production is completed. The chamber for this type of reaction has recently undergone revision as shown in Figure 3.3.7 (Massey and Lien 1979). The design, on the principle of a cyclone separator, allows entry and mixing of fission products such that the entire mixture is spinning around the sides of the vessel. This greatly increases the surface area, so the hydrides and H_2 can readily escape into the exit volume. From use of high-speed photography it appears that reaction zones occur (Lien 1978). This type of vessel has given the highest yield of hydrides obtained to date. It also has allowed the most rapid and efficient degassing of noble gases from the original fission products, to the extent that the degassing cycle can be decreased to only 100

to 200 ms, compared to the original 600 ms required using the system illustrated in Figure 3.3.3.

4. EXAMPLES OF CURRENT RESEARCH

4.1 Studies of Isotopes Important to the Decay Heat Problem

From both a technological and a fundamental scientific standpoint the decay properties of the short-lived antimony nuclei are important. Technologically, in designing a power reactor provisions must be made for several categories of hypothetical accidents. For example, if an accident causes the loss of the cooling water, automatic controls would instantly drop the reactor control rods back into the core, thereby stopping operation of the reactor and any further fission-product production. However, residual fission products holding a large amount of energy would remain. This energy would be converted into heat at a rate governed by the half-lives and by the decay energies of all the fission products present.

To design emergency cooling systems to compensate for this heat production, one approach is to identify those short-lived isotopes that produce the decay heat and then to calculate the rate of heat production. We have examined the contributions and their uncertainties from individual isotopes produced from ^{235}U fission products for a burst irradiation and for cooling times (t_c) of 10 and 100 s. For t_c of 10 s, more than 97% of the total decay power is due to about 84 isotopes. By summation of individual contributors, 26 isotopes account for 94% of the total energy variance. Data on these nuclei are extremely useful for the flexible and efficient design of next-generation reactors. These data include the properties of the short-lived antimony isotopes 4.2-min $^{132}Sb^m$, 2.4-min $^{132}Sb^g$, 2.3-min ^{133}Sb, and 10-s ^{132}Sb (Prussin et al. 1974).

From a fundamental point of view these same isotopes are important to understanding the nature of the shell model at large isospin and the

interaction of nucleons. Lately it has been possible to circumvent a number of earlier calculational problems in use of the shell model by application of the Lanezos algorithm that tridiagonalizes in an iterative fashion large, sparse matrices (Whitehead 1972; Whitehead et al. 1977; Sebe and Nachimkin 1969; Talman 1964; Hausman 1977; also see Appendix I).

The study of the ^{132}Sb, $^{132}Sb^{m}$, ^{133}Sb, and ^{134}Sb has been performed over a period of several years and includes singles, $\gamma\gamma$-coincidence, $\gamma\gamma t$-coincidence, and multiscale spectroscopy using the LLL autobatch facility. Studies of the shorter-lived 10-s ^{134}Sb decay were performed on the improved facility. The former experiments used an ion exchange resin to concentrate the antimony for measurement (Ishimori and Nakamura 1963; Ishimori, Nakamura, and Nurakami 1961) while the latter required a different catcher because the kinetics of ion exchange were too slow to be effective for the study of the 10-s activity. For the 10-s ^{134}Sb experiments, coincidence measurements required between 10 and 15 working days to acquire the requisite 25×10^{6} events for proper evaluation.

These investigations have shown the previously-documented average gamma-ray energy data to be incorrect by as much as 45%. For example, the changes in average γ-ray energies of isotopes associated with reactor-safety decay-heat isotopes are:

Value	Isotope	
	^{133}Sb	^{134}Sb
$E\gamma$, MeV/fission (previous work)	1.874	2.086
$E\gamma$, MeV/fission (previous work)	2.531	2.632
Percent change	45	26

The structure of ^{134}Te previously known from studies using ISOL systems is shown in Figure 4.1.1. Because of lower statistics available from this type of experiment only the first four levels of ^{134}Te were known. Also, the branching intensity of the 6_2^+ to 4_1^+ level was unknown. As shown in Figure 4.1.2 the data from chemical isolation not only can identify several higher-lying levels but also can provide a measure of the 6_2^+ to 4_2^+ transition. If we make the assumption that the M1 component does not compete in a significant manner, this transition suggests that the valence nucleons beyond shell closure fail to replicate the transition rates of the nuclear level decays. One approach is to use our measured value of the $6_2^+ \rightarrow 6_1^+$ (B) vs $6_2^+ \rightarrow 4_1^+$ (A) (Figure 4.1.2) transition rates that is more than an order of magnitude different from the predicted value (see inserted bar graph in Figure 4.1.2), using a simple model (calculation I). By including excitation of the core nucleons it is possible to account for the experimental results (calculation II). A more realistic approach, of course, is to use this transition-intensity ratio to set the effective M1 operator for this region (Heyde and Meyer, unpublished).

The lower-energy-level properties of ^{132}Te that result from the decay of $^{132}Sb^g$ and $^{132}Sb^m$ are shown in Figures 4.1.3 and 4.1.4. It should be noted that between $^{132}Sb^g$, $^{133}Sb^m$, and ^{133}Sb, decay over 500 γ rays can be identified. These give levels up to 5 MeV in ^{132}Te. When the decay properties of the higher-lying levels are combined with the levels shown, the suggested J^π values are obtained as compared to the results of our calculations in Figure 4.1.5. Of particular interest is the observation of the 8^+ and 10^+ level suggested by Sistemich (1978) whose properties subsequently were calculated by Heyde and Sau (1979). It should be noted, however, that the γ rays associated with the decay of these levels are only a fraction of the total γ-ray intensity at that energy in the decay of $^{132}Sb^g$ and $^{132}Sb^m$. The level structure of ^{132}Te to approximately 3 MeV is shown in Figure 4.1.6 and compared to that calculated by our large-scale

shell-model code. (A short description of these calculations is given in Appendix I.) It should be noted that the unusual deexcitation behavior of the second 2^+ level at 1665 keV in ^{132}Te is consistent with other Te nuclei as shown in Figure 4.1.6.

4.2 Studies of Nuclei Near N = 50 and Possible Coexistence of Deformed States in N = 49 Nuclei

A second type of work using chemical isolation systems deals with processes at high excitation energies. Using chemical techniques, Tomlinson (Tomlinson 1966; Tomlinson and Hurdus 1965, 1967, 1968) first showed that the As and Sb fission products possessed beta-delayed neutron emitters as predicted earlier (Keepin 1958). The measurement of neutron spectra with high resolution ^{3}He ionization chambers (Shalev and Cuttler 1973) has led to the discovery of spectra with pronounced peak structure with spacings that are large compared to level-density calculations (Franz et al. 1974; Kratz 1978). Their work discussed later at this conference (Kratz 1979), was based on neutron and high-energy γ-ray spectra measurements of chemically-isolated ^{85}As (2 s), ^{87}Br (55 s), and ^{135}Sb (1.8 s) (Nuh 1977; Franz et al. 1974; Shihab-Eldin et al. 1977; Kratz 1978). The conclusions concerning the spectral shape and beta-intensity distribution are quite different from those arrived at by Hardy, Jonson, and Hansen (Jonson et al. 1976) and by Gjøttrud, Hoff, and Pappas (1978) based on a purely statistical model approach (Hansen 1974). More detailed statistical model calculations of Prussin, Oliviera, and Kratz (1979) show that it is not possible to obtain simultaneous agreement between the general shape of the neutron energy spectra and the experimental data on neutron branching ratios to individual levels in the daughter nucleus such as populated in the $^{85}\text{As} \xrightarrow{\beta^-} {}^{85}\text{Se}^* \xrightarrow{n} {}^{84}\text{Se}$ sequence. Important to their work is a precise knowledge of the ^{84}Se-level structure available to the beta-delayed neutron emission of $^{85}\text{Se}^*$. Only five levels at 1455, 2122, 2700,

3541, and 5159-keV were known when investigations into the decay of ^{84}As to levels of ^{84}Se were undertaken.

The decay of 5-s ^{84}As was studied by simultaneous measurement of high-energy multiscaled singles and three-parameter ($\gamma\gamma$t) coincidence spectroscopy. The total chemistry time using the LLL-autobatch system was 1.8 s (Lien et al. 1979). Approximately 10,000 separations were required to accumulate 25 million coincidence events in approximately 10 working days. This allowed the identification of 28 levels below 4.75 MeV and the evidence of levels up to 9.5 MeV in ^{84}Se. These levels are in agreement with other N = 50 nuclei and similar to the N = 34, Z = 28 nucleus.

When we perform experiments on the decay of ^{84}As, we simultaneously accumulate data on the 14- and 19-s isomers of ^{82}As, as well as 13.3-s ^{83}As. Our initial analysis of data gives evidence for the levels of ^{82}Se shown in Figure 4.2.1. In Figure 4.2.2 we show the low-energy decay of 13.3-s ^{83}Se. The levels below 2300 keV that we observe in ^{82}Se, ^{83}Se, and ^{84}Se are shown in Figure 4.2.3.

Our data combined with ^{82}Se(d,p)^{83}Se reaction cross-section data suggest coexisting one-particle two-hole, deformed like structures and hole-core excitations in ^{83}Se with 49 neutrons. Recently we have been able to account for the properties of low-lying levels in the Z = 49 indium nuclei using a unified description (Heyde and Saw 1979). For the odd-mass indium nuclei we were able to describe a rich variety of nuclear phenomena by taking into account the coupling of both single-hole and one-particle two-hole configurations with the quadrupole and octupole vibrations of the underlying core nucleus. In particular we were able to account for the occurrence of low spin level with strong particle character rather than the expected strong hole character. We observe the population of levels in ^{83}As decay to the N = 49 nucleus ^{83}Se similar to those observed in the Z = 49 In nuclei. The levels at 963-, 1062-,

and 2076-keV that are populated by the ^{83}As decay also have been shown to have strong $d_{5/2}$ particle strength in the ^{82}Se(d,p)^{83}Se reaction studies (Lin 1965, Montestruque et al. 1978). Further investigation into the properties of these levels of ^{83}Se as well as similar levels identified in the lighter odd-mass Se nuclei are necessary before detailed tests can be made.

4.3 Exploration of Spin Traps with Spin Filters

There is a major effort today in research using heavy ion accelerators. Among the types of experiment being pursued is that of (HI, xnγ) where γ-ray spectroscopy is used to explore high-energy deexcitation toward the Yrast line and spin traps. An assumption in this type of work is that the majority of the deexcitation is to levels of lower spin, as shown in Figure 4.3.1. One may legitimately ask, is this an entirely correct assumption? A disadvantage the (HI, nγ) experimenters face is that large amounts of angular momentum are brought into the deexciting system. What is needed is a mechanism for entering a nucleus at a high excitation energy but at low to moderate spin. Then a nucleus with a known spin trap at lower energy could be used as a collector of events that increased in spin. Such experiments are possible using the spin filter technique. This technique, unfamiliar to those who have too long only focused on the exclusive use of reaction techniques has been used for a number of years under the guise of beta-decay scheme research (Wood 1978). Nuclei at high excitation energy but lower spins are very accessible using rapid automated nuclear chemistry on fission products. For example, as discussed in Section 4.2 we have observed levels in ^{84}Se up to 9.5 MeV from the decay of ^{84}As. The question of whether the beta decays are purely statistical (Hansen 1974) or exhibit nonstatistical effects (Kratz 1979; Prussin, Oliviera, and Katz 1979; Klapdor and Wene 1979) are yet to be explored in detail. Regardless, we are guaranteed of knowing that a distribution of low-spin states is being populated from these spin filters.

We have selected a spin filter of $11/2^-$ via the decay of $^{133}Te^m$ in order to explore the question of γ-ray deexcitation where the γ-ray cascade might go toward levels of increasing spin ("spin flow" increases in spin). The $^{133}Te^m$ daughter nucleus ^{133}I has a known 9-s $19/2^-$ isomer at 1634 keV, and the parent ^{133}Te has a decay energy of 3300 keV. Thus, if increasing spin flow occurs to an appreciable extent, this isomer ought be populated. Our initial experiment used ion-exchange chromatography to hold the Te fission products while eluting away the daughter iodine activity (see Figure 4.3.2). This allowed detailed singles and coincidence experiments to be performed (Meyer 1979). The resultant decay scheme indicated that approximately 12% of all $^{133}Te^m$ beta decays end up at the $19/2^-$ isomer via γ-ray cascades from levels higher in energy than the isomer. To prove that the isomer was indeed populated required a delayed measurement of the 9-s daughter ^{133}I activity. This was accomplished using the SISAK technique in the arrangement shown in Figure 4.3.3. The detection of the 9-s activity γ rays in the organic-phase spectra confirmed the existence of the population of the $19/2^-$ isomer in the decay from the $11/2^-$ spin filter $^{133}Te^m$ (Griffin, Skarnemark, and Meyer 1979). In Figure 4.3.4 we show the level scheme up to the isomer as populated by the $11/2^-$ spin filter $^{133}Te^m$ and in Figure 4.3.5 we show the percent γ-ray cascade from higher energy levels to the spin trap (isomer).

Further evidence of the occurrence of unexpected increasing spin flow is not necessarily confined to near shell closure. Recent studies at Mainz (Denschlag 1977; Walters 1978) on fission yield values for isomer ratios have shown that for a given element the ratio can change drastically from isotope to isotope. For example, isomer ratios in ^{99}Nb were found to be 0.068 for (σ high spin/σ high spin + σ low spin) a value far from 0.81, the value predicted by simple theory. On the other hand, the similar ratio for ^{97}Nb is 0.99, or a factor of 15 different.

The general spin flow and its rate, near but slightly greater in energy than the Yrast line, ought be explored further. Certainly, we should suspect that the state purity of levels should increase as the Yrast line is approached. Then, there ought be reasonable probability that a particular level could have as its major deexcitation increasing spin flow. This, in turn, would pump larger than expected angular momentum into a Yrast cascade resulting from a particular reaction. Again, this could partly be addressed far from shell closure by studying cases similar to the $^{133}Te^{m}$ spin filter by RANC studies of fission products.

4.4 Ground State Correlations and Unique First Forbidden Beta Decay in the ^{132}Sn Region

Our studies of odd-mass nuclei in the ^{132}Sn region have allowed us to develop a systematic body of information on unique first forbidden (ulf) beta decay. This type of beta decay is governed by the single matrix element $\langle \vec{\sigma} \cdot \vec{\tau}_1^2 \rangle$. As a consequence, the related f_1t value can provide information about the nature of the initial and final states connected by ulf beta decay. As seen in Figure 4.4.1 the log f_1t value for ulf beta decay between $7/2^+$ ground states and daughter $11/2^-$ levels ranges from the lower limit of 8.5 to above 10. The log f_1t value of 8.51 for the ulf beta decay of the single-proton-plus-closed-shell $^{133}_{51}Sb_{82}$ to $^{133}_{52}Te_{81}$ is now the fastest known ulf beta decay observed to date and represents the results of two independent measurements (Denschlag 1979; Meyer 1979). It has been suggested that the $11/2^-$ levels in these nuclei can be accounted for by a simple $h_{11/2}$ configuration. Thus, thi. large range of values must arise from effects associated with ground-state cluster correlations. If this is correct we should expect the fastest ulf decay for $^{133}_{51}Sb_{82}$ with only a single proton beyond the shell closure, and the slowest ulf decay for $^{133}_{53}I_{82}$ where the

three-proton cluster effect should be the strongest. As is shown in Figure 4.4.1, this is what is observed. Calculations are in progress to account for these variations on a quantitative basis (Paar and Meyer 1979).

5. SUMMARY

The field of rapid chemistry is itself entering a period of rapid expansion. Fast chemical research by Herrmann (Herrmann and Denschlag 1969; Trautmann and Herrmann 1976) has led to a large number of rapid chemical separation techniques that are now lending themselves to high degrees of automation. Recognition that gas-phase chemistry (Zendel 1978; Zendel et al. 1978) can provide both rapid delivery and virtually instantaneous chemistry may lead to the accessibility of a large class of studies. The advent of small high-speed centrifuges (H-10 of MEAB; Reinhardt 1979; Rydberg et al. 1979) that allow rapid-phase separation is providing the possibility of continuous and rapid solvent extraction systems (Trautmann 1978).

APPENDIX I

SHELL MODEL CALCULATIONS AND STUDY OF ^{132}Sn REGION AT LLL*

S. M. Lane, E. A. Henry, and R. A. Meyer

Microscopic calculations in the ^{132}Sn region for nuclei having additional nucleons or nucleon holes generally treat ^{132}Sn as an inert core. Difficulty arises in that the number of nuclei whose structure can be determined by conventional shell model techniques is limited. This is because of our inability to accommodate a sufficient number of orbitals. For example, the $1g_{7/2}$, $2d_{3/2}$, $2d_{5/2}$, $3s_{1/2}$, and $1h_{11/2}$ subshells comprise the "gddsh" major shell and provide 32 distinct orbitals for excitons of ^{132}Sn. When we consider the $^{133}_{51}Sb_{82}$ nucleus with a single valence proton and limit the states accessible to that proton to the major shell mentioned above, we have to account for 32 separate states. Next, consider the one-valence proton, one-valence neutron-hole nucleus, ^{132}Sb where we must deal with $32^2 = 1024$ multiparticle states. Finally, to consider the two-valence proton, two-valence neutron-hole nucleus, ^{132}Te requires that we allow for $(32 \times 21/2)^2$ = 246,016 states. Although there are various schemes whereby the appropriate choice of representation partially diagonalizes the Hamiltonian matrix, it is obvious that as more excitons are considered, the number of multiparticle states rapidly overwhelms the available computing power. Many ways have been discovered to avoid this problem and still describe the structure of spherical nuclei in the ^{132}Sn region, including the effective interaction models that describe the nucleus with collective coordinates, and the use of a combination of collection and independent particle models (where the motion of a few (one

*Part of this work constitutes the thesis of S. M. Lane to be submitted to the faculty of the University of California at Davis.

or two) valence nucleons is coupled to the correlated motion of the nuclear surface).

It is possible to circumvent these problems in still another way and retain the attendant advantages of a microscopic calculation. This can be accomplished by the application of a numerical method of diagonalizing very large vector spaces. The Lanczos algorithm tridiagonalizes in an iterative fashion large sparse matrices and does so in such a way that the lowest eigenvalues converge fastest. Following the method of Whitehead (Whitehead 1972, 1977) Hausman and Bloom at Lawrence Livermore Laboratory (Hausman 1977) have developed a computer program for solving the nuclear shell model eigenvalue problem that makes use of the Lanczos method. By employing an m-scheme multiparticle basis description, they dispense with coupling formalisms involving seniority and coefficients of fractional parentage. This makes it possible to cast the algorithms in a vector form suitable for execution on parallel processing computers. The program presently has the capacity to handle 30,000 multiparticle-basis states and 59 single-particle states. In a valence space consisting of the full gddsh shell, with the restriction that neutron-holes are excluded from the $1g_{7/2}$ subshell, detailed properties of nuclei having as many as five excitons may be calculated in this way.

We have applied the method of Whitehead as expanded by Hausman to nuclei with up to 5 excitons in the ^{132}Sn region as shown in Figure AI.1. The potential well in which the valence particles moved was approximated by a spherical harmonic oscillator well and matched in size. The interaction between valence nucleons that was used was one due to Petrovich, McManus, and Madsen (1969). This force, which is a modified form of the realistic Kallio-Kolltveit interaction (Kallio and Kolltveit 1964), has been used successfully for structure calculations in other regions of spherical nuclei. The

single-particle energies were obtained from the ^{132}Sn one neutron-hole spectrum (the $1h_{11/2}$ energy was readjusted in a few cases). When it is kept in mind that these calculations were made without the freedom of adjustable parameters, the agreement with experiment is found to be surprisingly good.

The wavefunctions we calculate for the two-proton two-neutron-hole nucleus, ^{132}Te, are given in Tables AI.1 and AI.2. Figure AI.2 shows the experimental level structure and the theoretical shell-model results for the 2-particle nucleus ^{134}Te. By contrast Degrieck and Vanden Berghe (1974) used up to three proton states of the ^{130}Sn core coupled to the motion of two valence protons in the Degrieck calculation. The protons had access to the entire gddsh shell and the residual interaction between them was the surface delta interaction. The SPE's, the single particle-phonon coupling parameter, the amplitude of the core oscillations, and the strength of the surface delta function were obtained by fitting the known energy levels of A = 126-134 Te energy levels. Only positive parity levels were calculated. Agreement with experiment is good, which is not surprising given the number of adjustable parameters.

Agreement between experiment and the calculation done here is also good. The shell model, however, predicts two low-lying 0^+ states not entirely confirmed experimentally as yet. It may be that these levels are not populated strongly in a ^{132}Te nucleus produced in beta decay.

Of particular interest are the model wavefunctions of the levels below 2.4 MeV. The valence protons remain paired in the $g_{7/2}$ subshell in all of the dominant configurations in this energy region. This implies that the low-energy ^{132}Te-level structure is similar to the ^{130}Sn two-neutron hole structures. This is indeed the case as seen in Figure AI.3 where negative parity and 0^+, 1^+, and 2^+ ^{132}Te levels are compared to the ^{130}Sn levels. Configurations greater than 20% are also shown.

TABLE AI.1. $^{132}_{52}Te_{80}$ Positive Parity Model Wavefunctions

Energy		% Configuration													
(keV)	J	$g^2d_3^{-2}$	g^2h^{-2}	g^2s^{-2}	$g^2d_5^{-2}$	$g^2d_3^{-1}s^{-1}$	$d_5^2h^{-2}$	$g^2d_5^{-1}d_3^{-1}$	$d_5^2d_3^{-1}s^{-1}$	$g^2d_5^{-1}s^{-1}$	gd_5h^{-2}	$d_5^2d_3^{-2}$	$gd_5d_3^{-2}$	$gd_5d_5^{-2}$	gd_5s^{-2}	Total
0	0^+	34	22	16	7	22										79
992	2^+	34	13	11		22										80
1454	0^+		38	25		7	12									82
1625	4^+	39	24	16	7	7										93
1698	0^+	42	5	35												82
1722	2^+	31	21	5		19										76
1746	6^+	38	24	16	7											85
2013	2^+	17	31			27	6									81
2052	2^+	39	31			8	6									84
2238	4^+		53			6	9	12								80
2279	1^+					82			5							87
2371	6^+		6								35		26	5	11	83
2478	0^+		19				33					20				72
2491	6^+		68				13									81
2501	1^+	23	6			44		8		12						93
2507	4^+	12	27			25		17								81
2556	10^+		74				14									88
2582	8^+		75				14									89
2671	2^+										32		25		11	68
2701	4^+	5	5								33		19		8	70

TABLE AI.2. $^{132}_{52}Te_{80}$ Negative Parity Model Wavefunctions

Energy	J	% Configurations				Total
		$g^2d_3^{-1}h^{-1}$	$d_5^2d_3^{-1}h^{-1}$	$g^2s^{-1}h^{-1}$	$d_5^2s^{-1}h^{-1}$	
1882	7^-	78	8			86
1994	5^-	40		41		81
2233	4^-	80	8			88
2326	6^-	72	7	11		90
2464	5^-	49		32		81
2505	6^-	20		62	6	88
2911	9^-	82		11		93

CALCULATION OF THE $^{134}_{52}Te_{82}$ LEVEL STRUCTURE

Figure AI.4 shows the calculated spectrum of the two valence proton nucleus $^{134}_{52}Te_{82}$ along with the experimental spectrum. The model wavefunctions are given in Table AI.3. We note that all of the low-lying positive-parity-level model wavefunctions are at least 80% pure and are members of the two multiplets arising from the couplings $|\pi lg^2_{7/2}>$, $|\pi\, g_{7/2}, \pi 2d_{5/2}>$, and $|\pi\, d^2_{5/2}>$. Total spins of 0^+, 2^+, 4^+, and 6^+ are obtained by coupling two $g_{7/2}$ protons. The anti-aligned (0^+) arrangement gives the maximum overlap and therefore the lowest energy, while the 2^+, 4^+, and 6^+ states have successively higher energies. The levels at 2328, 2575, 2656, 2699, 2715, and 2737 keV are due to a $|\pi\, g_{7/2}, \pi d_{5/2}>$ multiplet that can couple to total spins of 1^+, 2^+, 3^+, 4^+, 5^+, and 6^+. For short-range forces, deShalit and Talmi (1963) showed that the 6^+ state has the lowest energy, followed by the 4^+ state and the 2^+ state. Odd J states are shown to have very little splitting. Finally, the levels at 2368, 2737, 3158, and 3386 keV come from the coupling of two $d_{5/2}$ protons.

Figure AI.4 displays the results of shell-model calculations by Wildenthal and Larsen (1971) and by Baldridge (1977). Since both these groups have calculated properties of the N = 82 isotones, $^{134}_{52}Te_{82}$, $^{135}_{53}I_{82}$, and $^{136}_{54}Xe_{82}$, it will be interesting to compare their results with those obtained here. First, however, it will be useful to discuss the models they used.

Wildenthal's valence space consisted of the full $1g_{7/2}$ and $2d_{5/2}$ proton subshells with a single particle allowed in the $3s_{1/2}$ or $2d_{3/2}$ subshells. The nucleon-nucleon force was a modified surface delta interaction (MSDI) (Arvieu and Moszkowski 1966). The interaction strengths and the single-particle energies were obtained by a least squares fit to all of the then known positive parity levels in N = 82, A = 136 ≈ 140 nuclei.

TABLE AI.3. $^{134}_{52}Te_{82}$ Model Wavefunctions

Energy		% Neutron Configurations										
(keV)	J	g^2	d_5^2	d_3^2	s^2	gd_3	h^2	gd_5	g_5	d_5s	d_5d_3	Total
0	0^+	82	10	2	1		4					99
1338	2^+	96	1			1	1					99
1600	4^+	98						1				99
1704	6^+	97						3				100
2328	6^+	3						97				100
2368	0^+	14	80	4	2							100
2575	4^+	1				1		96	1			99
2656	2^+	1	1			2		96				100
2699	5^+							100				100
2715	3^+							100				100
2737	1^+							100				100
3158	2^+	1	85							5	2	93
3386	4^+		90						2		6	98

Baldridge, on the other hand, used a more sophisticated semirealistic residual interaction. The Brueckner G matrix (Brueckner 1955) from the Reid soft-core potential (Reid 1968) was taken as the zeroth order proton-proton interaction, and first-order phenomenological corrections were made by adding two-body pairing and multipole forces. The strengths of the correction terms were determined by fitting the $^{134}_{52}Te_{82}$ spectrum. The valence space consisted of the full gddsh proton shell. Systematic trends of levels having large single particle spectroscopic factors (Wildenthal and Larson 1971; Wildenthal, Newman, and Auble 1971) were used to obtain estimates of the SPEs.

The three calculations correctly predict the first four states in ^{134}Te, although this agreement is not particularly noteworthy in the Baldridge calculation because his correction-term strengths were determined by fitting these levels. Both the calculation done here and that by Baldridge correctly predict the second 6^+ level but there is disagreement as to the location of the 1^+ level. The leading components of the model wavefunctions obtained in the three calculations are compared in Table AI.4. The mixture of configurations is substantially the same for all three calculations, with the Wildenthal wavefunctions being slightly less pure.

TABLE AI.4. Comparison of Model Wavefunctions of $^{134}_{52}Te_{82}$

Energy[a]			% Proton Configuration							
(keV)	J	Calculation[b]	g^2	d_5^2	d_3^2	s^2	h^2	gd_3	gd_5	Total
0	0^+	L	82	10	2	1	4			99
		B	72	16	3		7			98
		W	81	18						99
1338	2^+	L	96	1			1	1		99
		B	98							98
		W	81	5			7			93
1600	4^+	L	98						1	99
		B	99							99
		W	86						7	93
1704	6^+	L	97						3	100
		B	98							98
		W	86						14	100
2328	6^+	L	3						97	100
		B							98	98
2737	1^+	L							100	100
		B							100	100

[a]Energies, given in keV, refer to this work.

[b]L refers to this work; B refers to Baldridge 1977; W refers to Wildenthal 1971.

TABLE AI.5. Proton Single Particle--Energies (MeV)

Orbit	Experimental	Baldridge	Wildenthal	This Work
$1g_{7/2}$	0.0	0.0	0.0	0.0
$2d_{5/2}$	0.963	0.963	0.88	0.910
$1h_{11/2}$	2.792	2.76		3.058
$2d_{3/2}$		2.69	3.12	2.597
$3S_{1/2}$		2.99	2.95	2.377

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Meyer R. A., Note 1

A note for the reader who does not come from a lineage of printers, as I do, and so is not accustomed to the Johannes Gutenberg syndrome of reading typeset backwards--zniam read backwards is MAINZ--which, as one will discover by reading further, has been responsible for much work in the field of rapid radiochemical isolation and studies.

Meyer R. A., Note 2

Dr. John Miskel (1979) has pointed out a delightful article by G. E. M. Jauncey (1946) that is highly recommended to the reader interested in the early history of radiochemistry research.

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MAE/ld

FIGURE CAPTIONS

FIG. 1.1. Relative output of an ISOL system ion source (after Wohn 1977).

FIG. 3.1.1. Plot of k_2 versus η (courtesy of P. C. Stevenson, Lawrence Livermore Laboratory).

FIG. 3.1.2. Plot of η versus k_1 (courtesy of P. C. Stevenson, Lawrence Livermore Laboratory).

FIG. 3.2.1. Generalized flow diagram for SISAK system.

FIG. 3.2.2. Schematic drawing of gas-jet system for continuous chemistry system.

FIG. 3.2.3. Internal parts of an H-10 centrifuge (A) labeled half section (B) isometric showing entry of mixed phases (orange) and exit of light (yellow) and heavy (red) phases (courtesy of J. Rydberg).

FIG. 3.2.4. Flow diagram for Tc separation at Mainz (courtesy of SISAK Consortium, Skarnemark 1979).

FIG. 3.2.5. Electrostatic deposition of activity from a SISAK experiment.

FIG. 3.2.6. Schematic drawing of gas-phase chemistry system for the isolation of Te from fission products (adapted from Zendel 1978 and Zendel et al. 1978).

FIG. 3.3.1. Schematic drawing of Mainz "building block" batch processor (adapted from Tittel et al. 1977).

FIG. 3.3.2. Time sequence for Lawrence Livermore Laboratory autobatch system.

FIG. 3.3.3. Time sequence of chemical steps in the Lawrence Livermore Laboratory autobatch system.

FIG. 3.3.4. Segments of spectra and schematic of As separation.

FIG. 3.3.5. Improvements to the Lawrence Livermore Laboratory autobatch facility.

FIG. 3.3.6. Original hydride generation chamber for Lawrence Livermore Laboratory autobatch processor.

FIG. 3.3.7. Half section for Cyclone separator.

FIG. 4.1.1. Level structure of ^{134}Te from ISOL studies.

FIG. 4.1.2. Level scheme of ^{134}Te derived from studies at Lawrence Livermore Laboratory using autobatch system (Henry 1979).

FIG. 4.1.3. Decay of ^{132}Sb^{g} to levels of ^{132}Te below 2650 keV.

FIG. 4.1.4. Decay of ^{132}Sb^{m} to levels of ^{132}Te below 3500 keV.

FIG. 4.1.5. Comparison of ^{132}Te levels derived from studies at Lawrence Livermore Laboratory and shell model calculations (see Appendix I and Lane 1979).

FIG. 4.1.6. Comparison of branching ratios of the second $J^{\pi} = 2^{+}$ level in even-even mass Te isotopes.

FIG. 4.2.1. Levels of ^{82}Se populated in the decay of ^{82}As.

FIG. 4.2.2. Levels of ^{83}Se populated in the decay of ^{83}As.

FIG. 4.2.3. Levels of ^{82}Se and ^{84}Se below 2300 keV.

FIG. 4.3.1. Relation of spin filter region to Yrast cascade.

FIG. 4.3.2. Isolation technique used for study of ^{133}Te.

FIG. 4.3.3. Chemical systems for isolating 9-s ^{133}I^{m} from fission product Te. Operation of the upper system is limited to some tens of minutes, because of Te migration to the 0.1 M H_2SO_4 phase. The lower system is more stable and is insensitive to the chemical state of the iodine (providing no I_2 is formed).

FIG. 4.3.4. Decay of ^{133}Te^{m} to levels below 9 s $19/2^{-}$ isomeric level (spin trap).

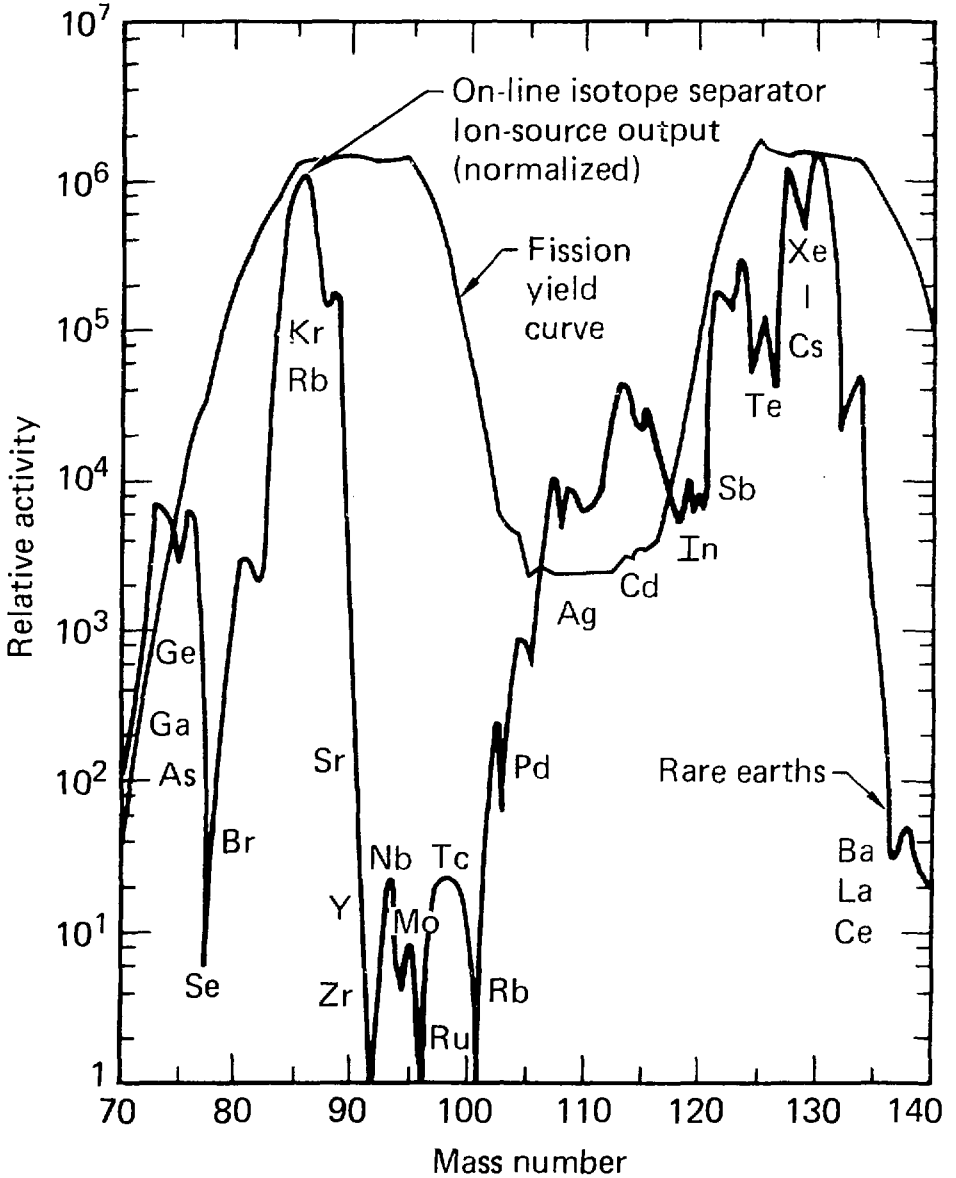
FIG. 4.3.5. Levels of ^{133}I that decay by γ-ray cascades of increasing spin.

FIG. AI.1. Nuclei calculated in the present model.

FIG. AI.2. Comparison of ^{134}Te levels calculated by shell model code to those found in fission-product experiments.

FIG. AI.3. Positive and negative levels of ^{132}Te compared to those of ^{130}Sn.

FIG. AI.4. Comparison of theoretical calculations of ^{134}Te levels.


On-line isotope separator
Ion-source output
(normalized)
Fission
yield
curve
Relative activity
Mass number
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Rb
Sr
Y
Zr
Nb
Mo
Ru
Tc
Rb
Pd
Ag
Cd
In
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Ce
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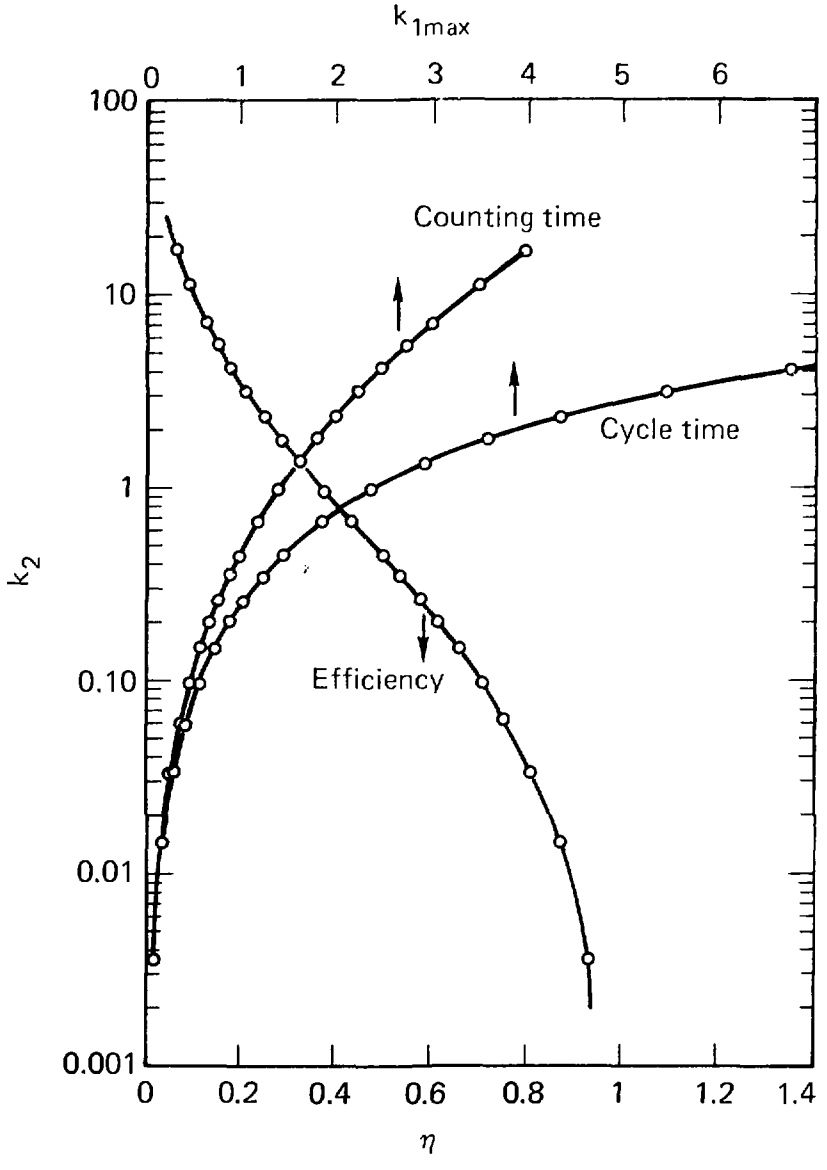
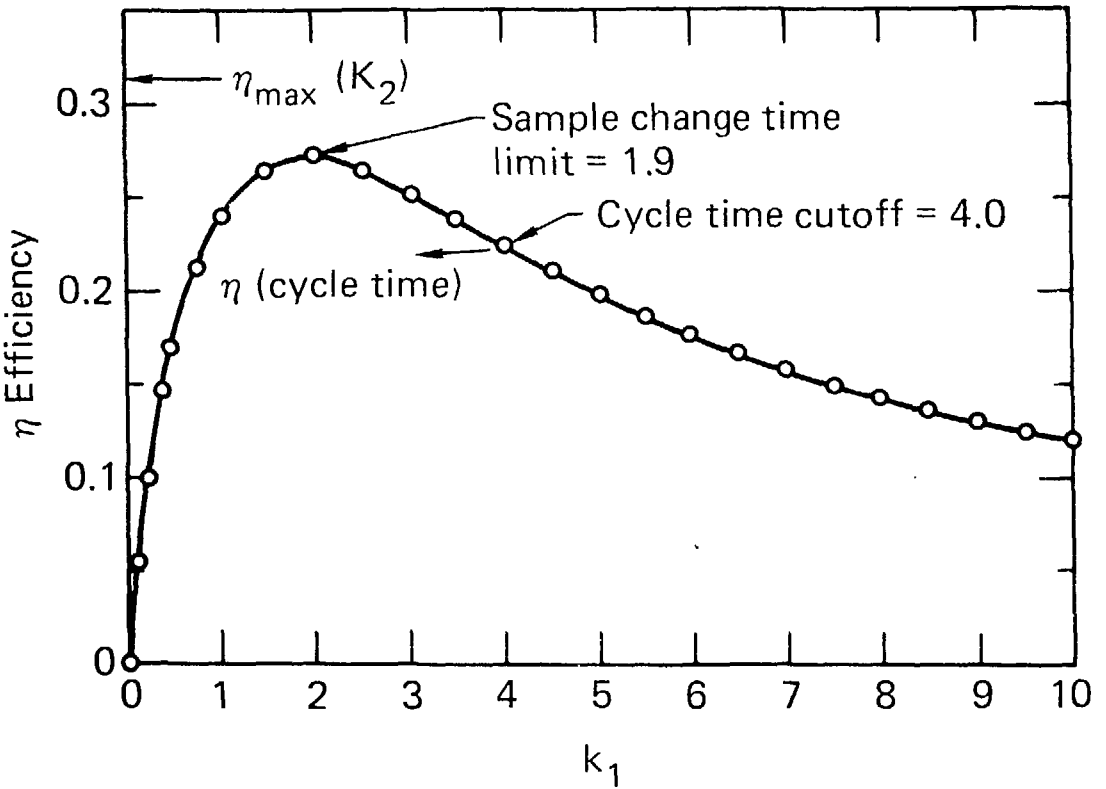
Ga
As
Se
Br

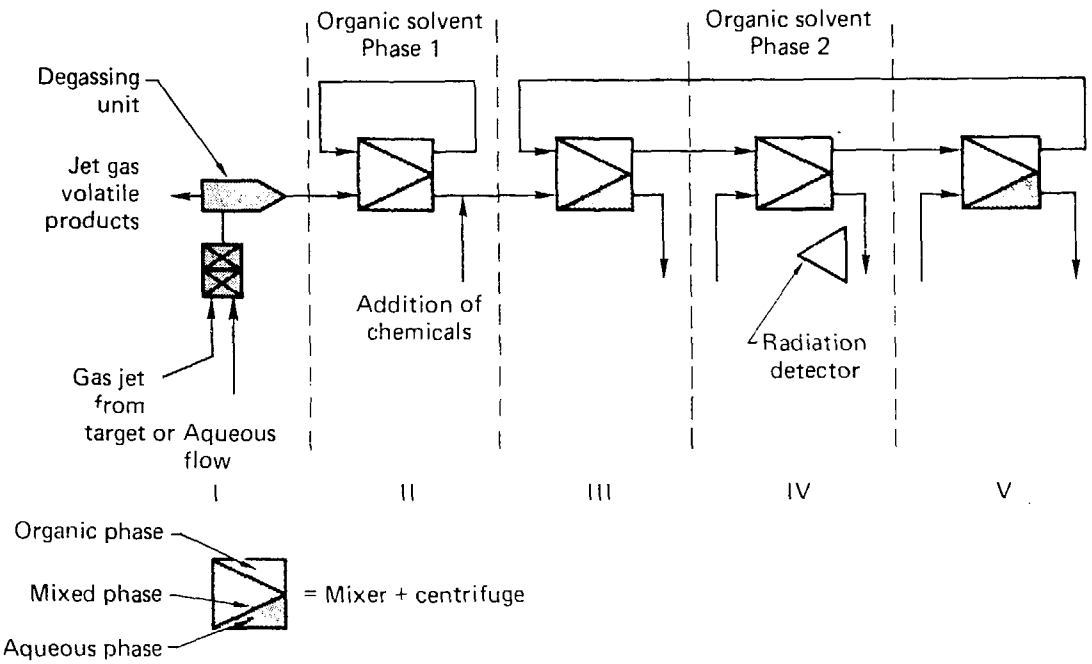
Meyer - FIG. 1.1



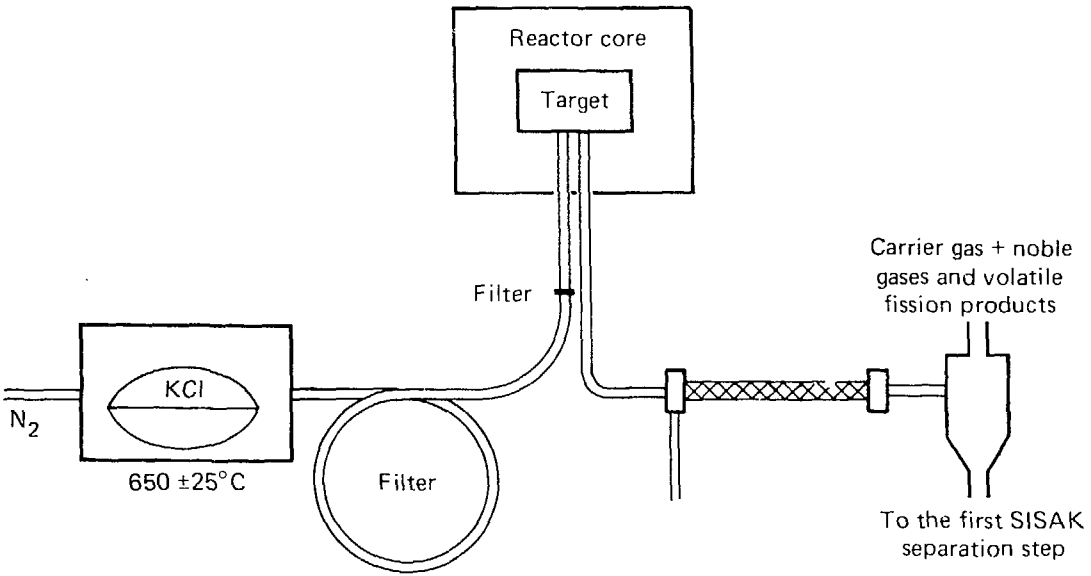
Meyer - FIG. 3.1.1



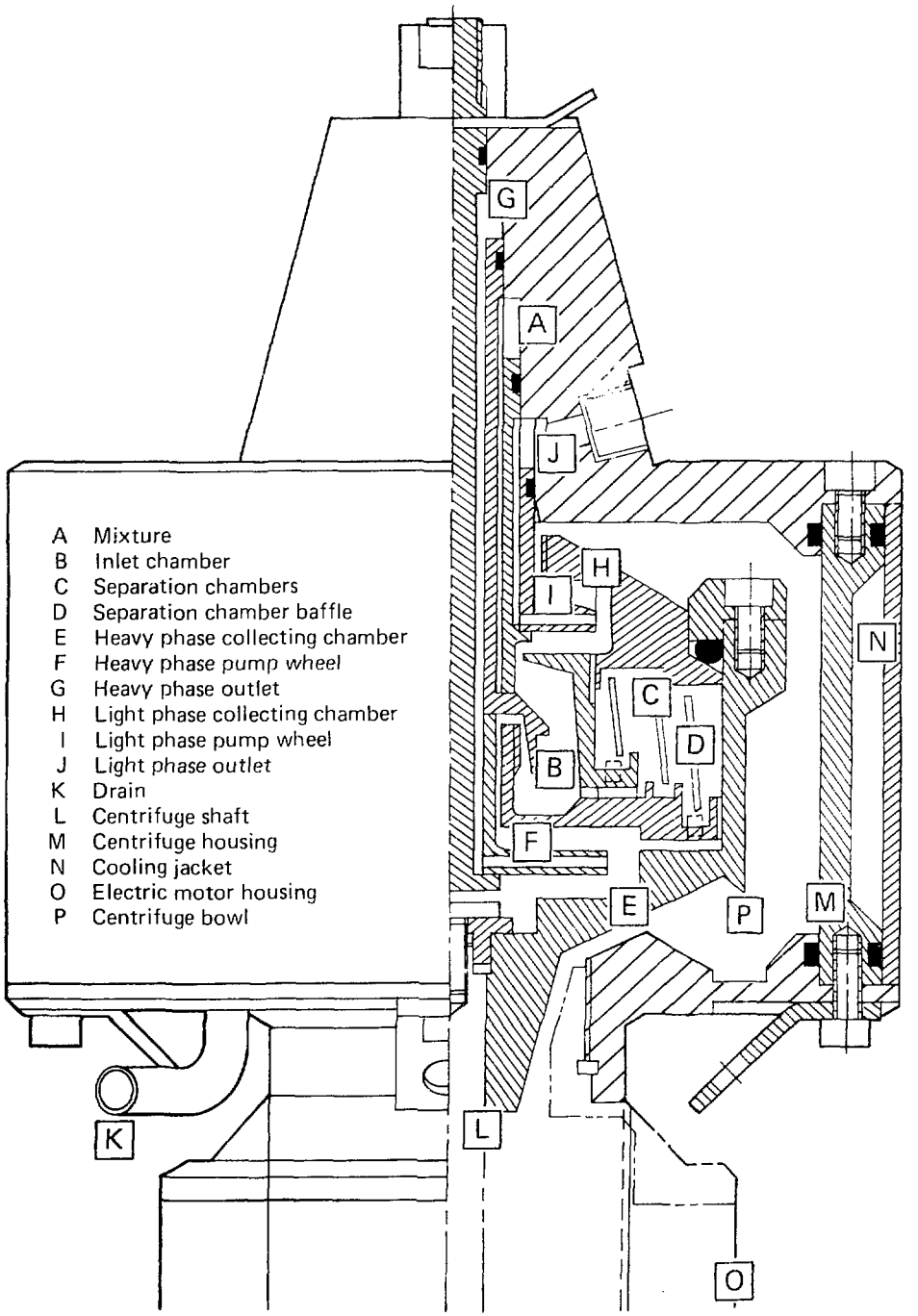
Meyer - FIG. 3.1.2



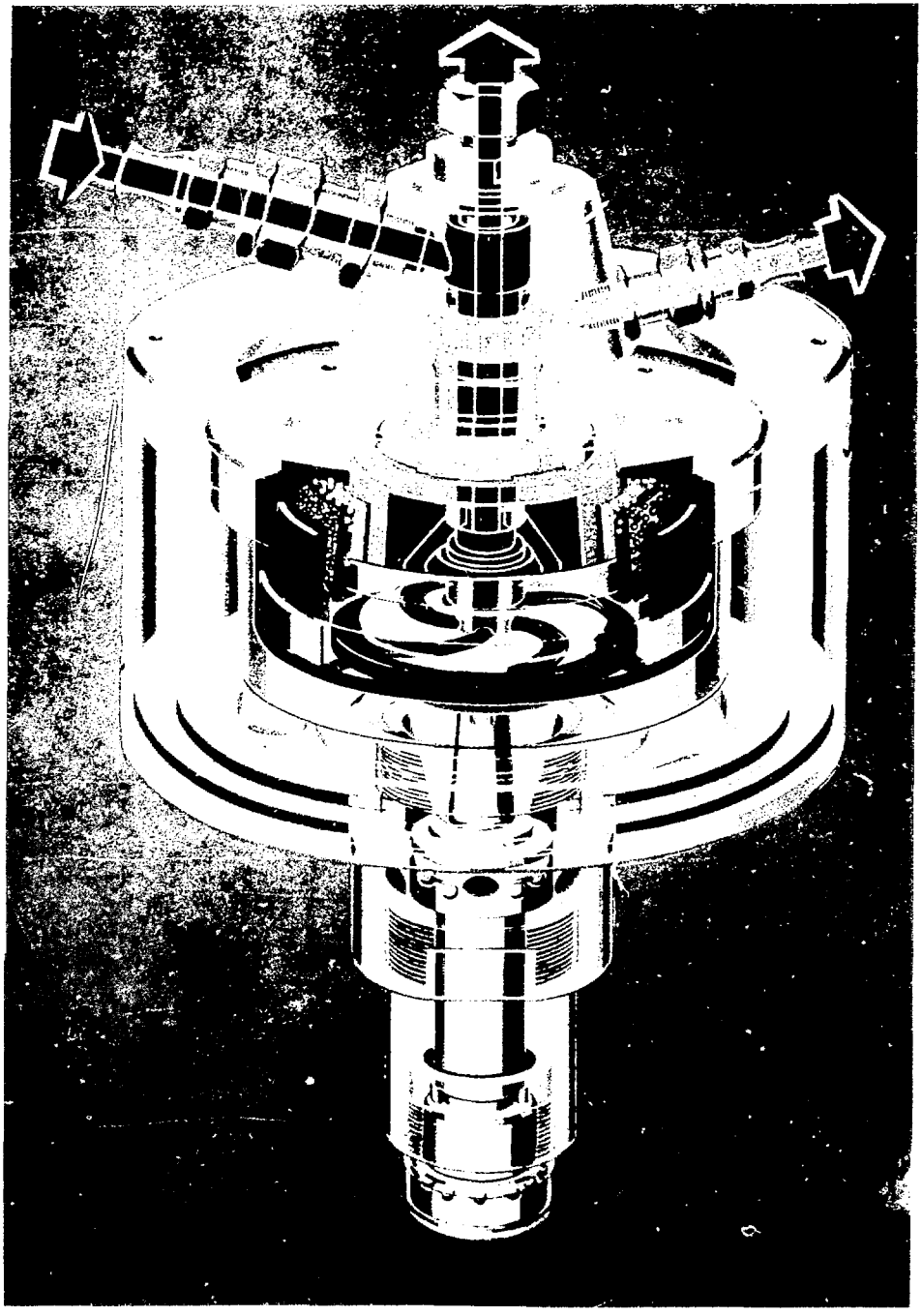
Meyer - FIG. 3.2.1



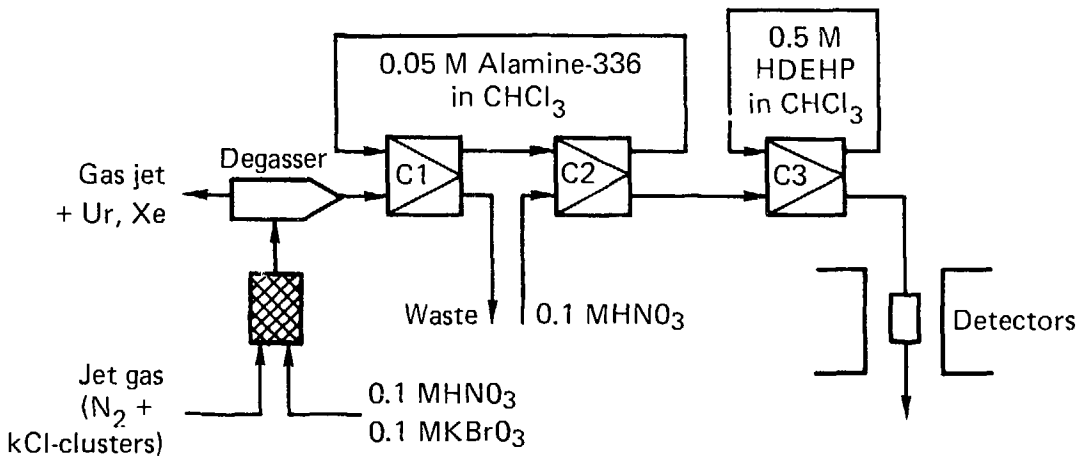
Meyer - FIG. 3.2.2



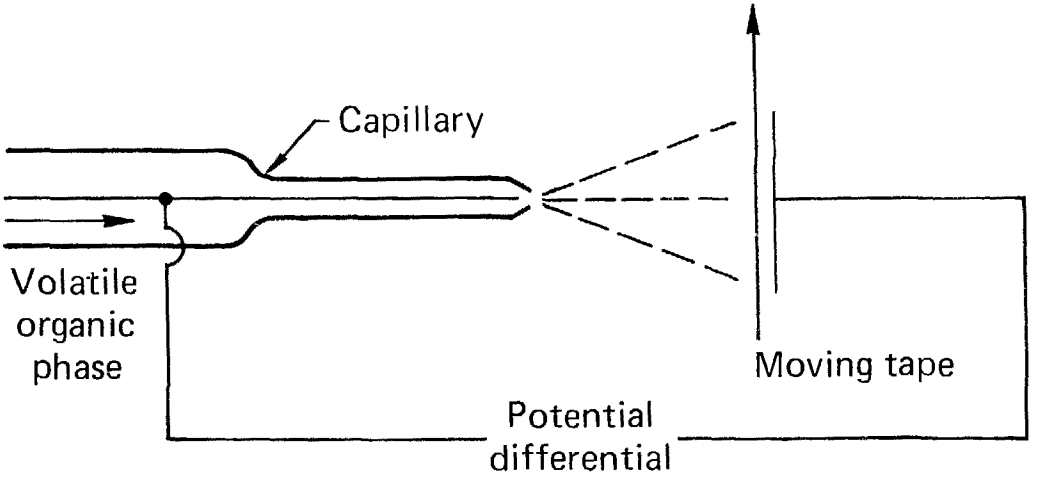
Meyer - FIG. 3.2.3A



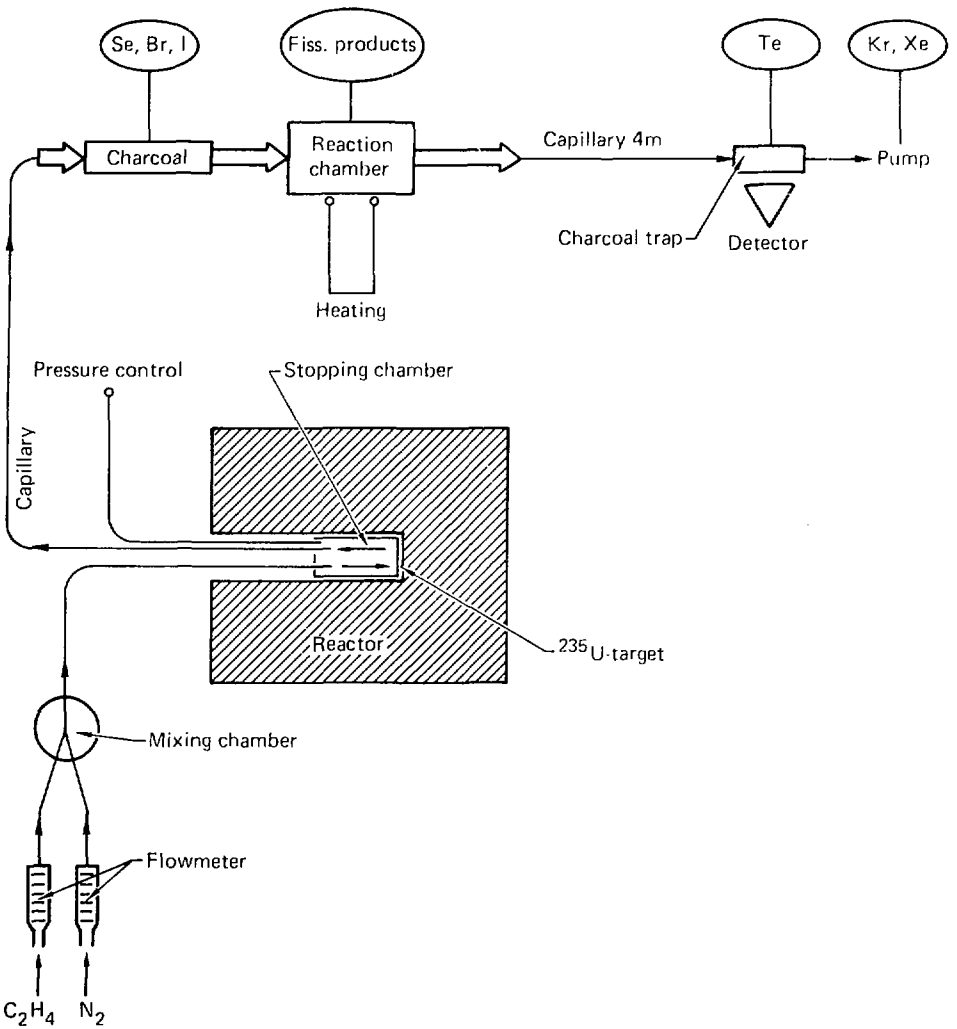
Meyer - FIG. 3.2.3B



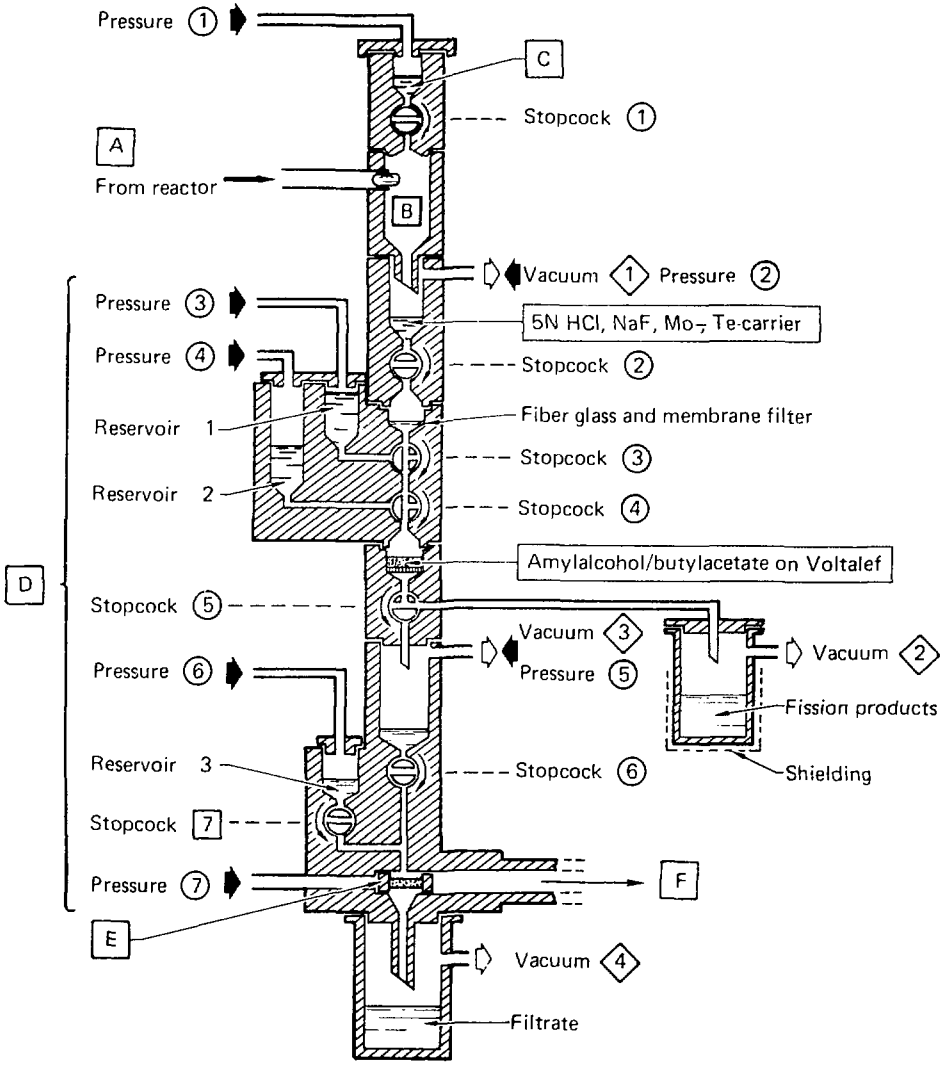
Meyer - FIG. 3.2.4



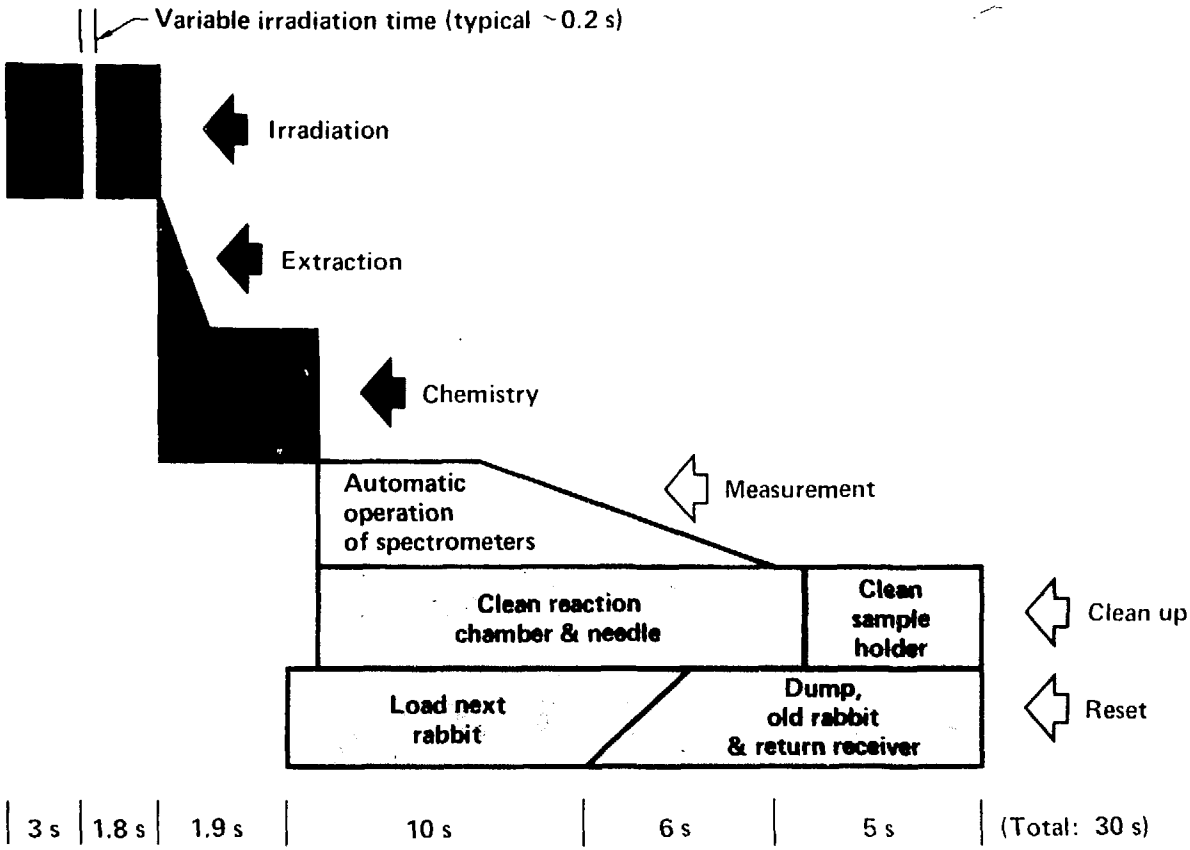
Meyer - FIG. 3.2.5



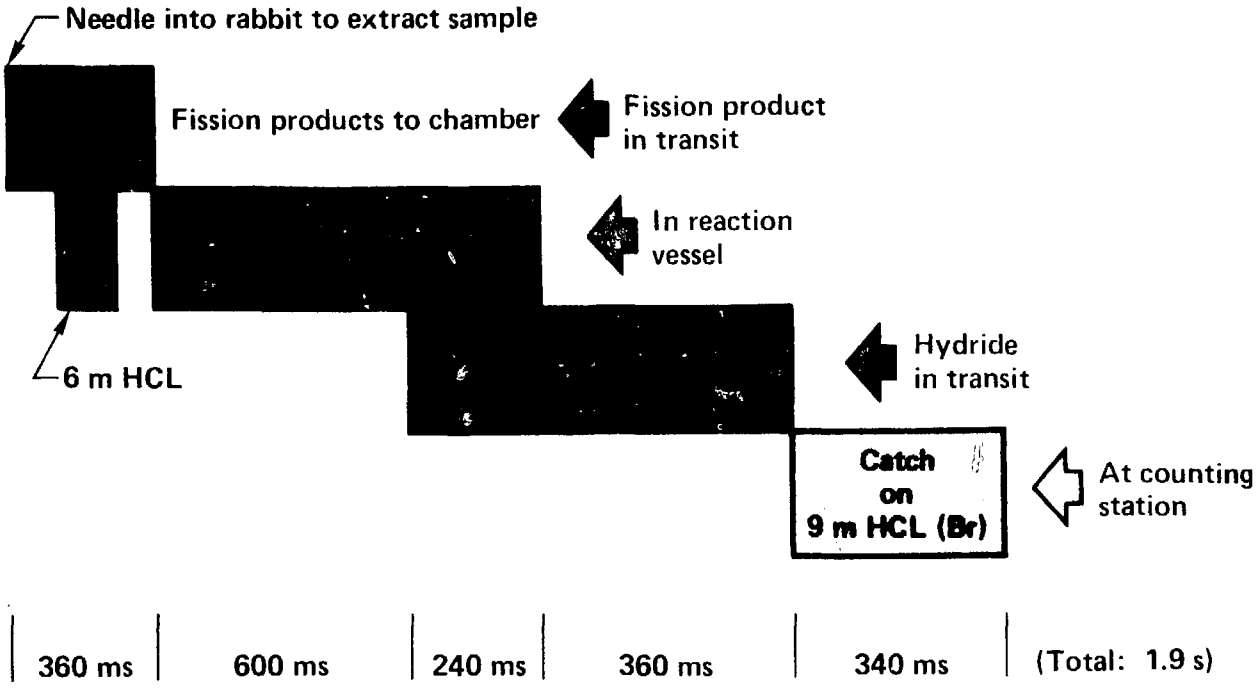
Meyer - FIG. 3.2.6



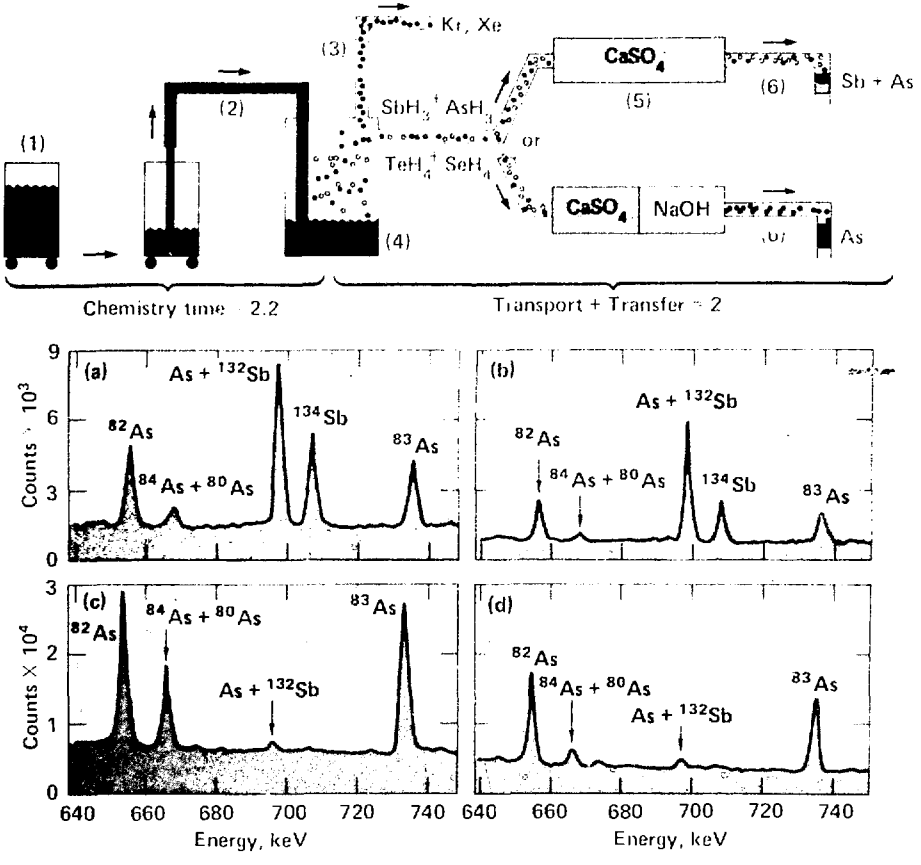
Meyer - FIG. 3.3.1



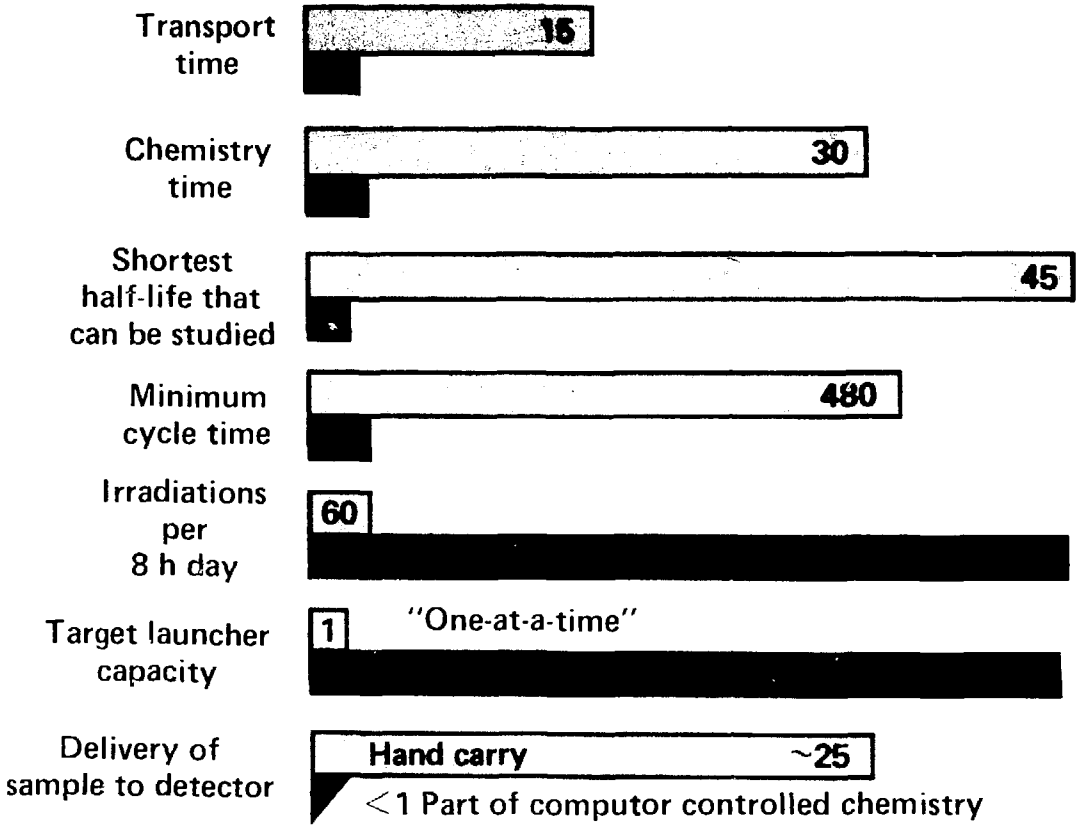
Meyer - FIG. 3.3.2



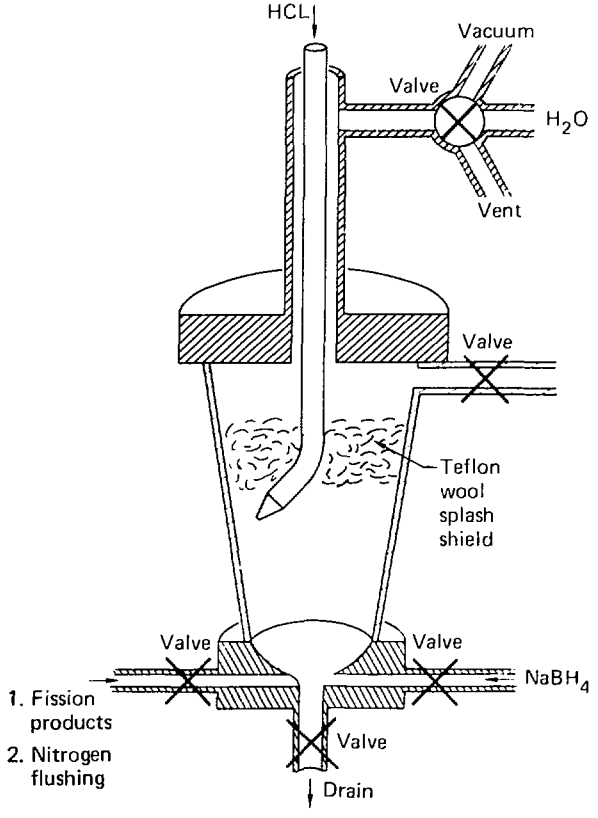
Meyer - FIG. 3.3.3



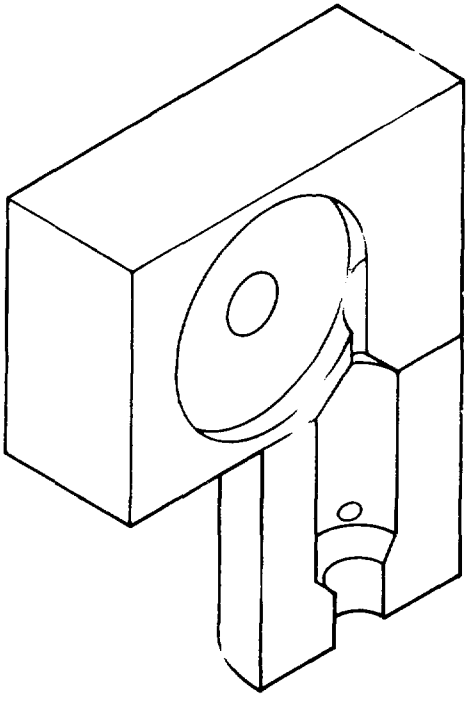
Meyer - FIG. 3.3.4



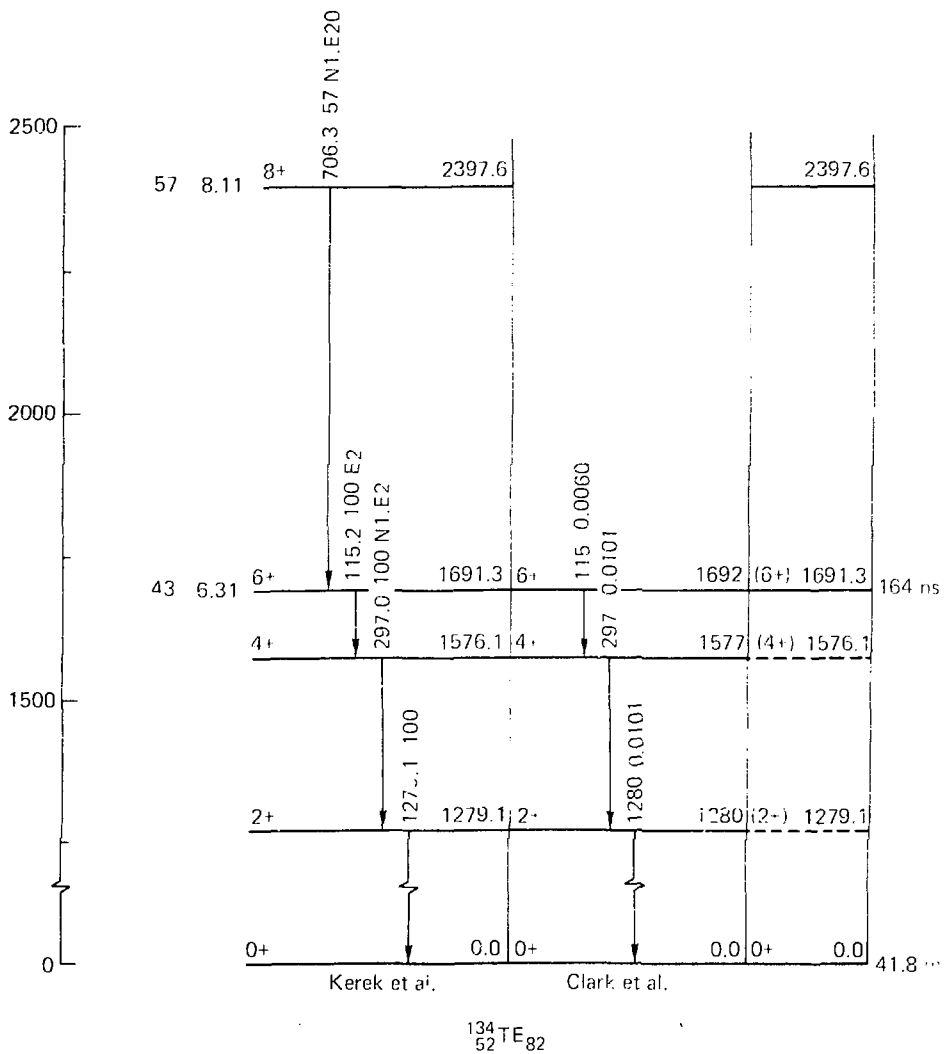
Meyer - FIG. 3.3.5



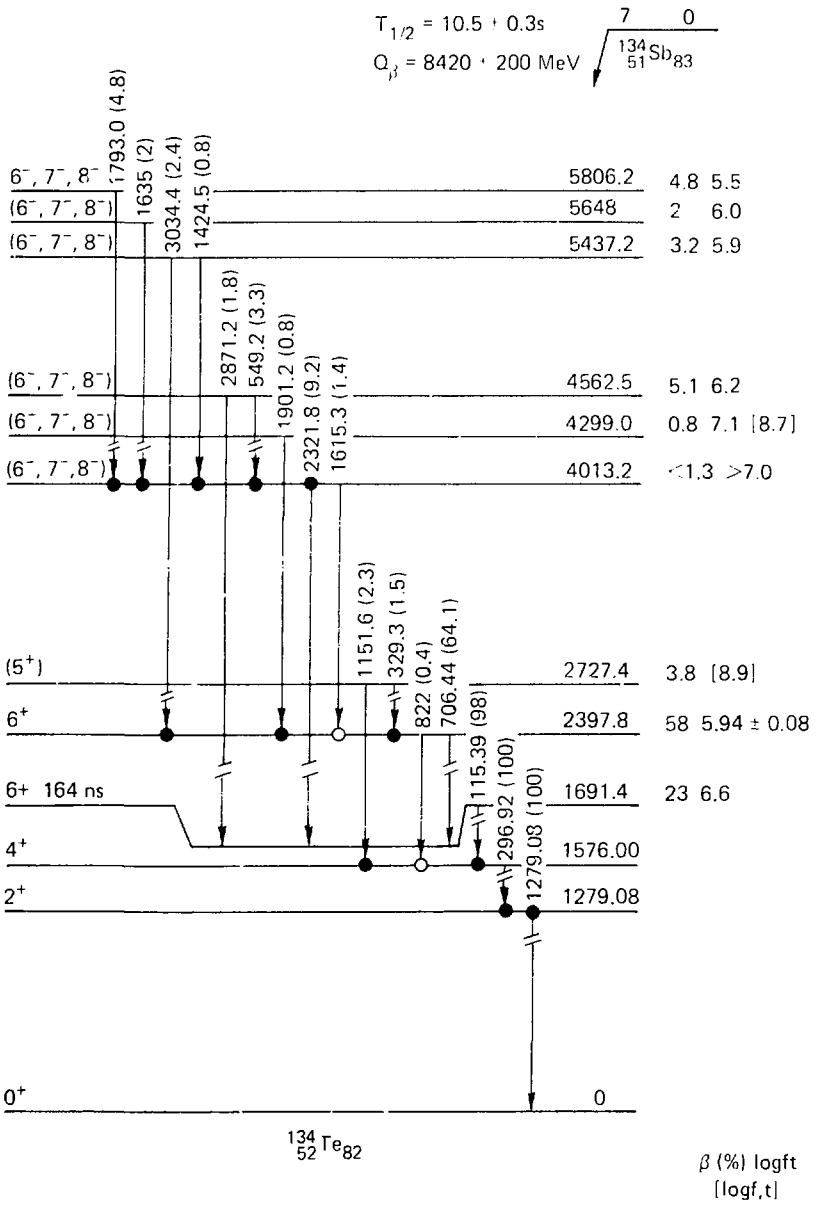
Meyer - FIG. 3.3.6



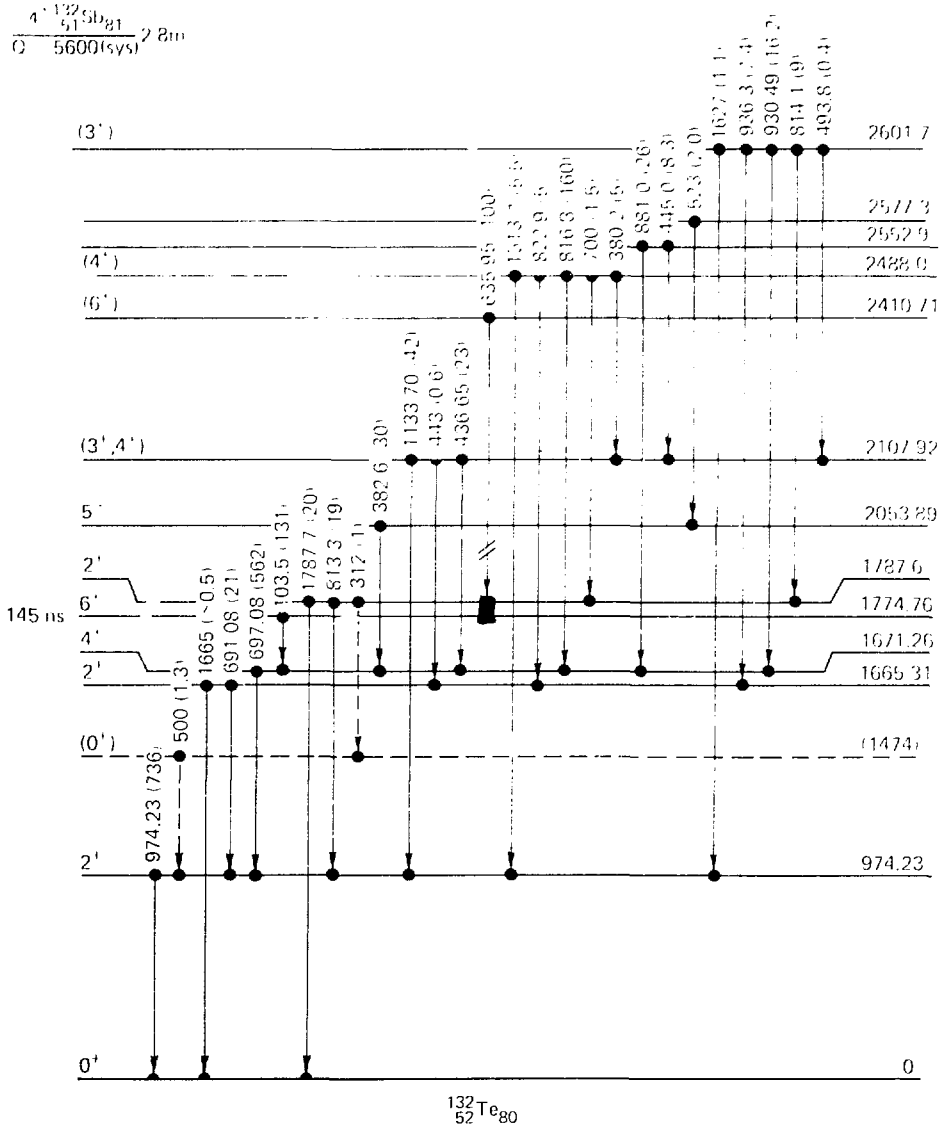
Meyer - FIG. 3.3.7



Meyer - FIG. 4.1.1

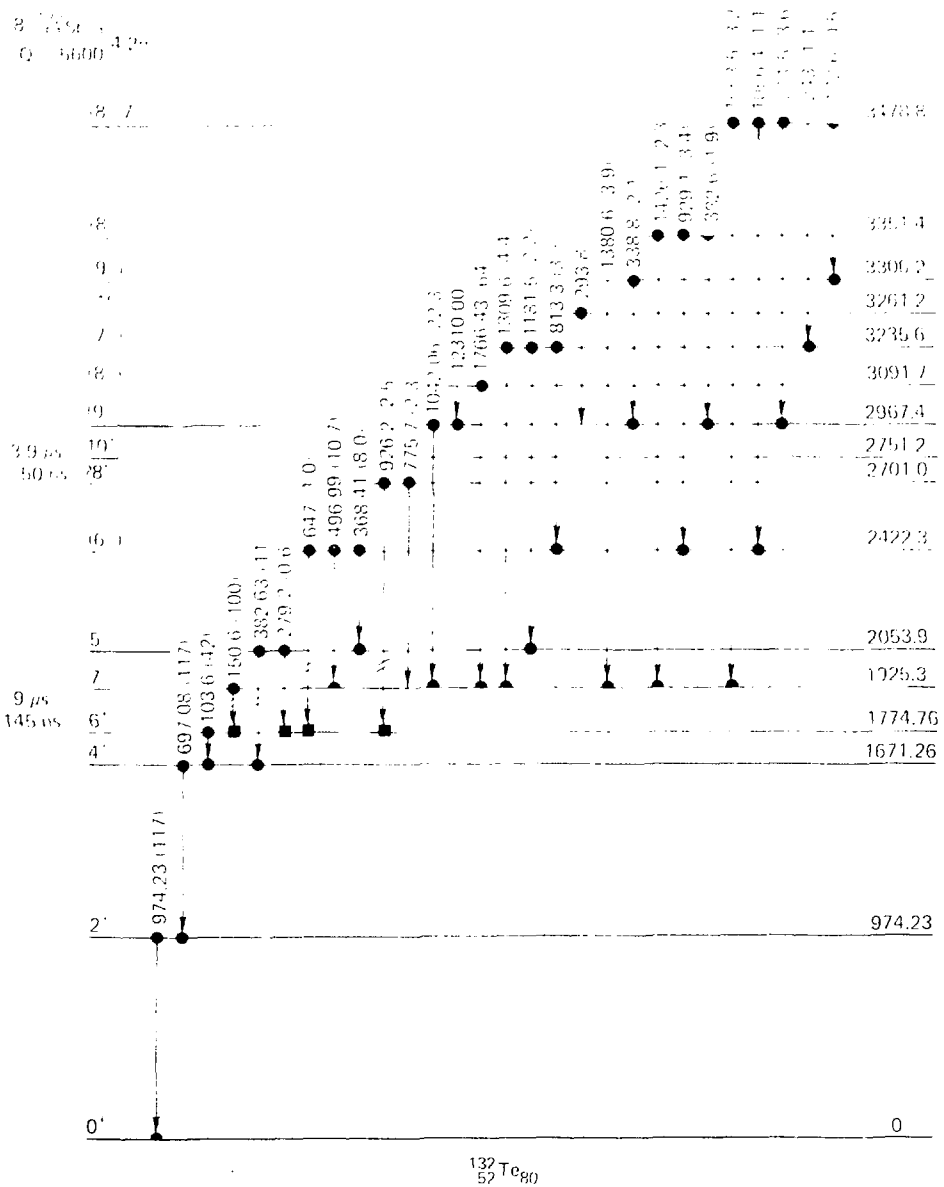


Meyer FIG. 4.1.2

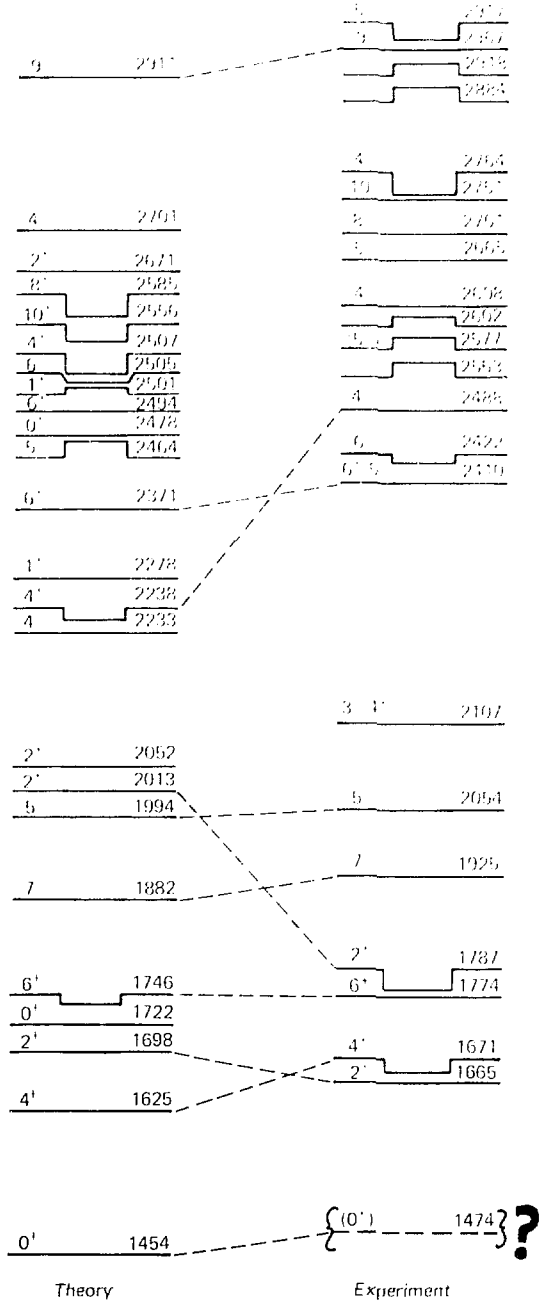


$^{132}_{52}Te_{80}$

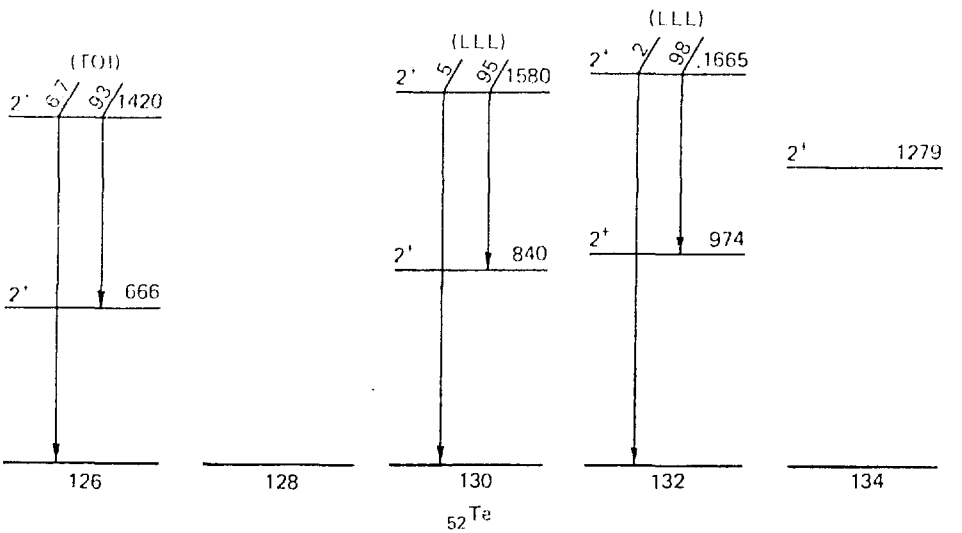
Meyer FIG. 4.1.3



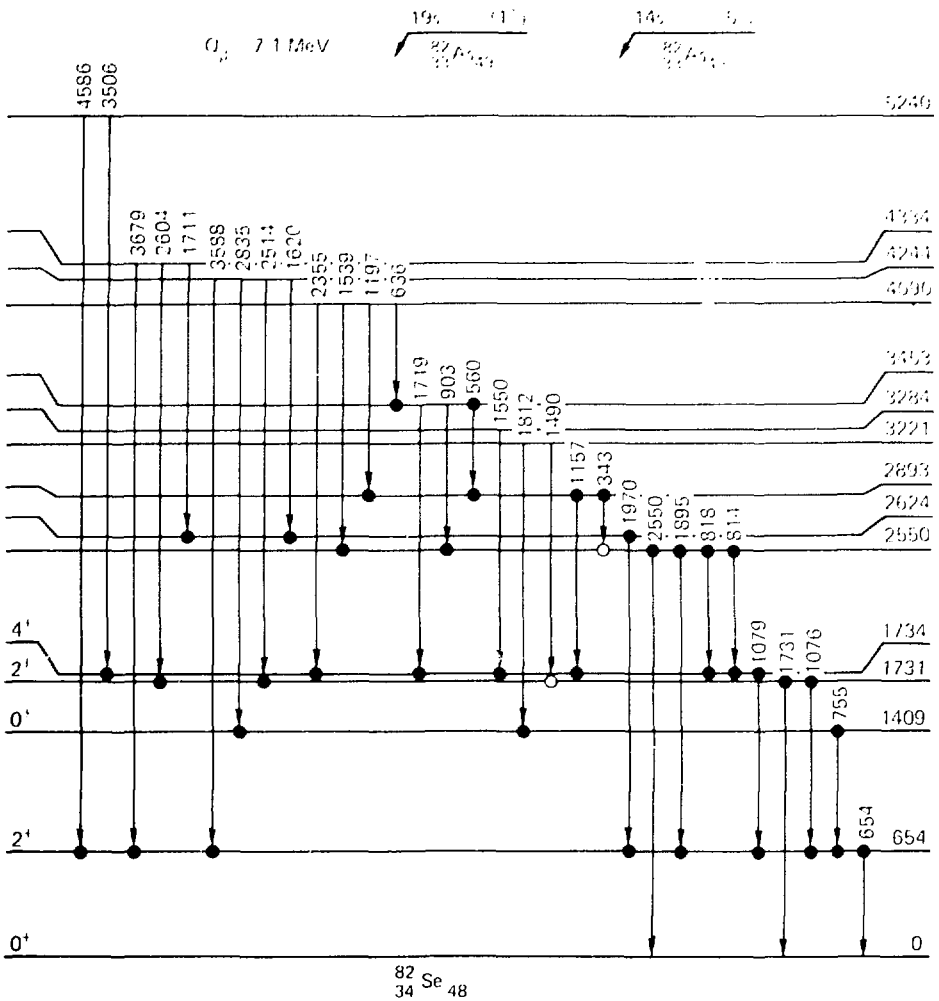
Meyer FIG. 4.1.4



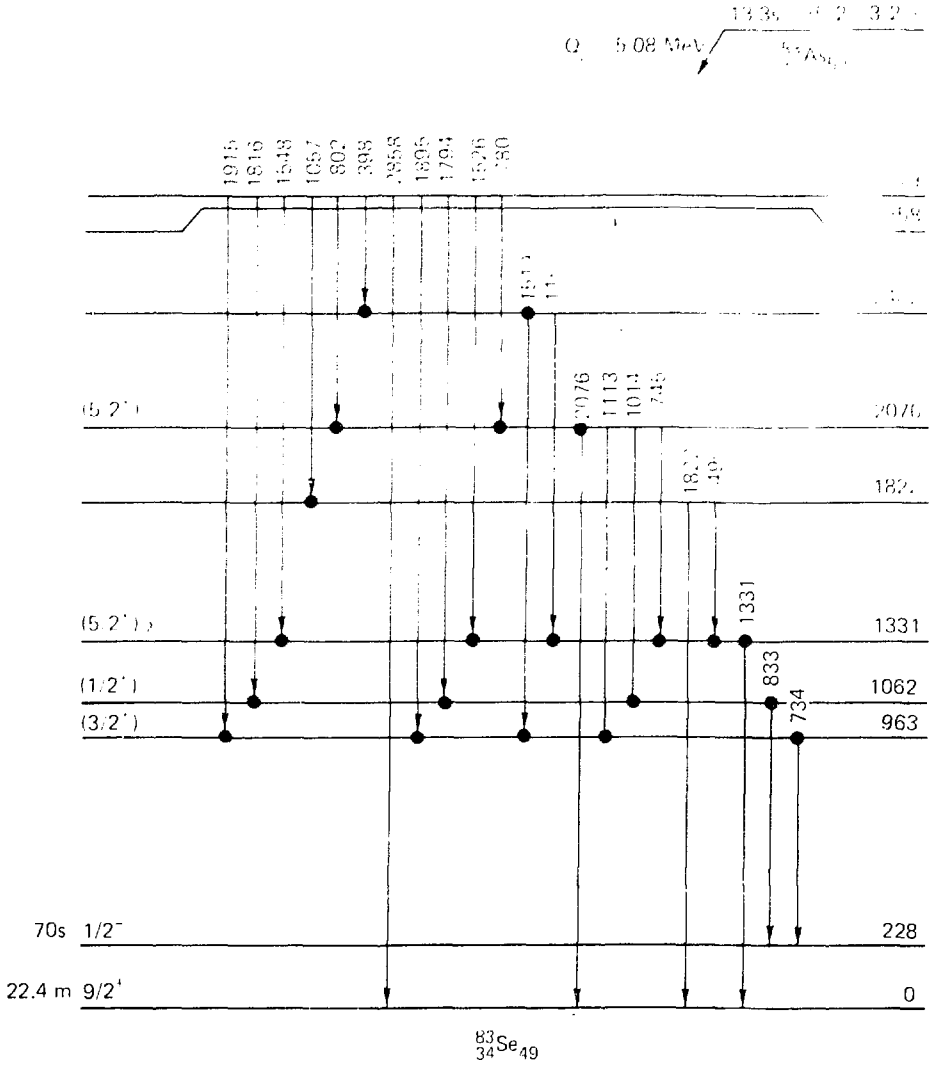
Meyer FIG. 4.1.5



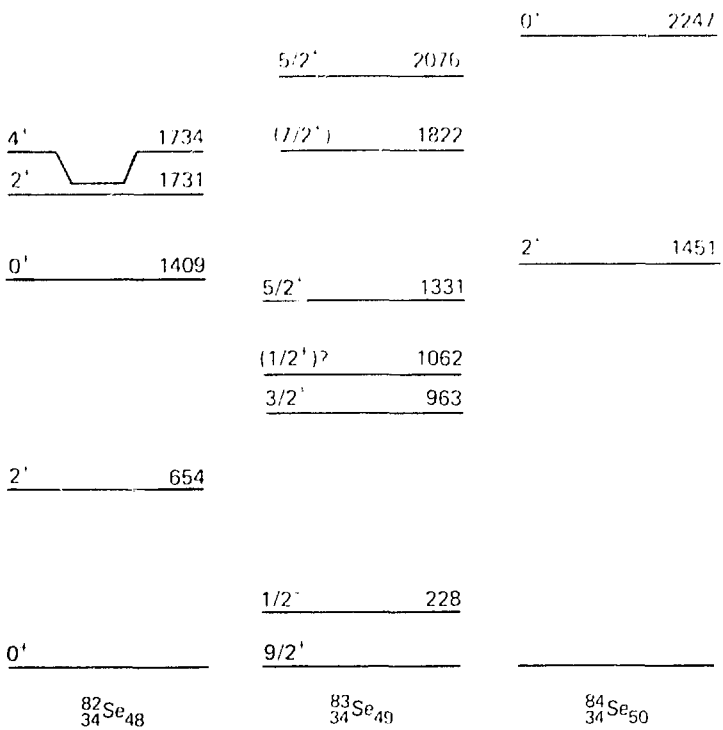
Meyer FIG. 4.1.6



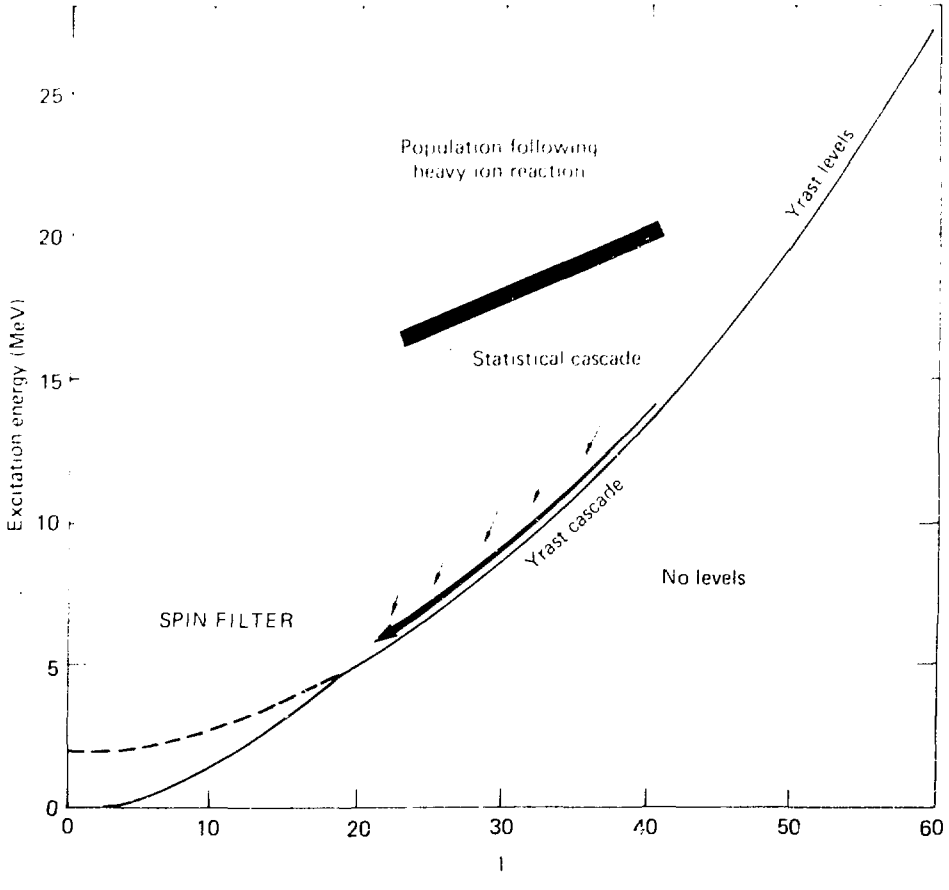
Meyer FIG. 4.2.1



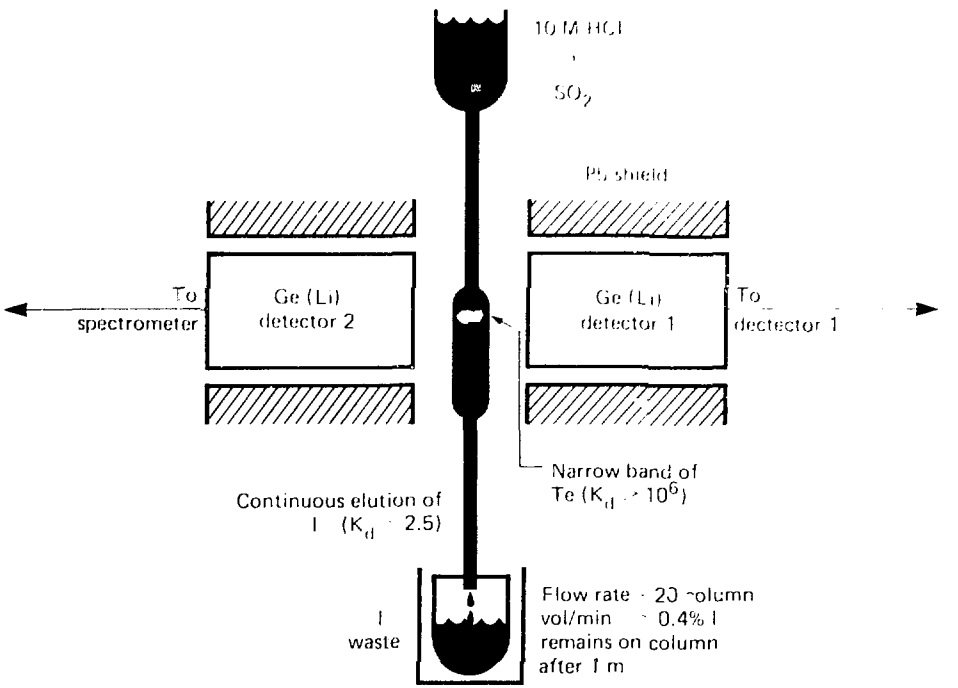
Meyer FIG. 4.2.2



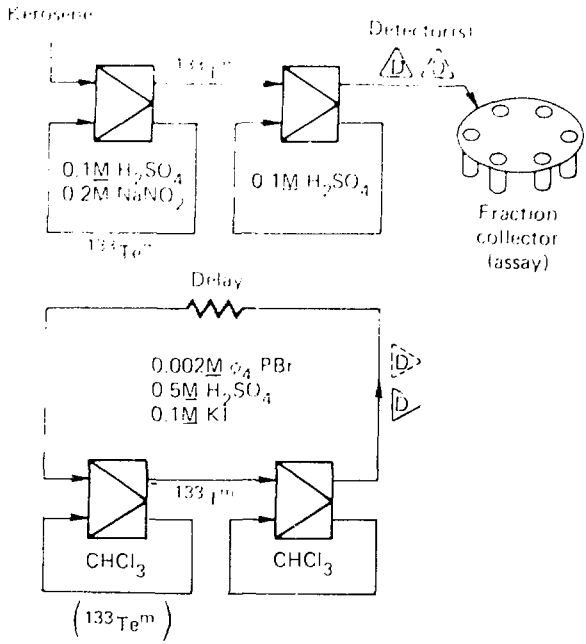
Meyer FIG. 4.2.3



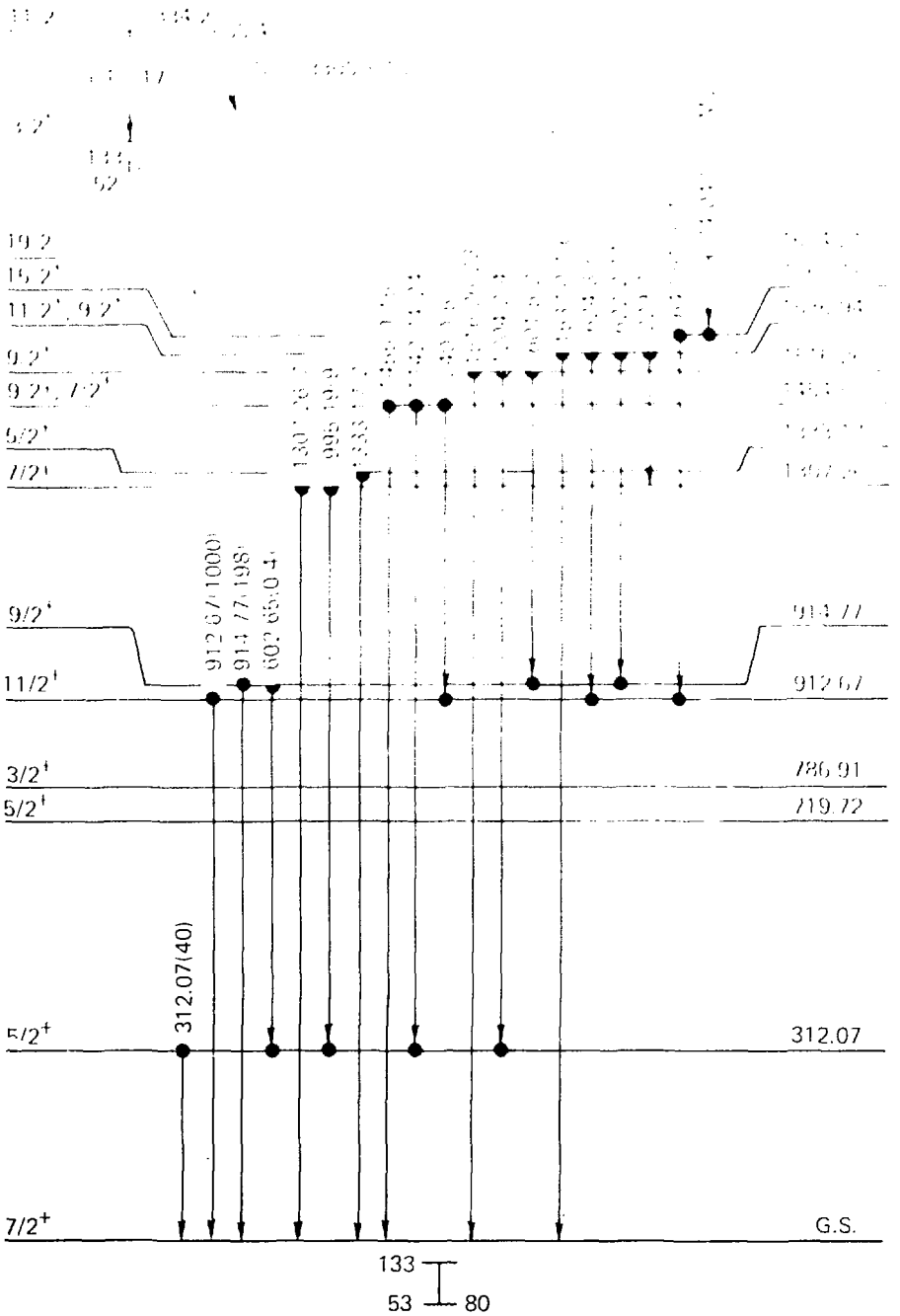
Meyer FIG. 4.3.1



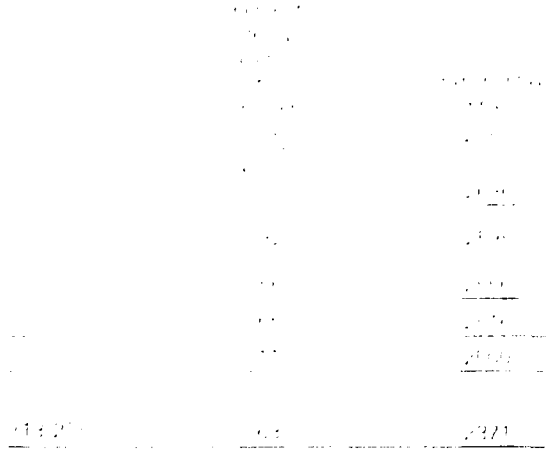
Meyer FIG. 4.3.2



Meyer FIG. 4.3.3



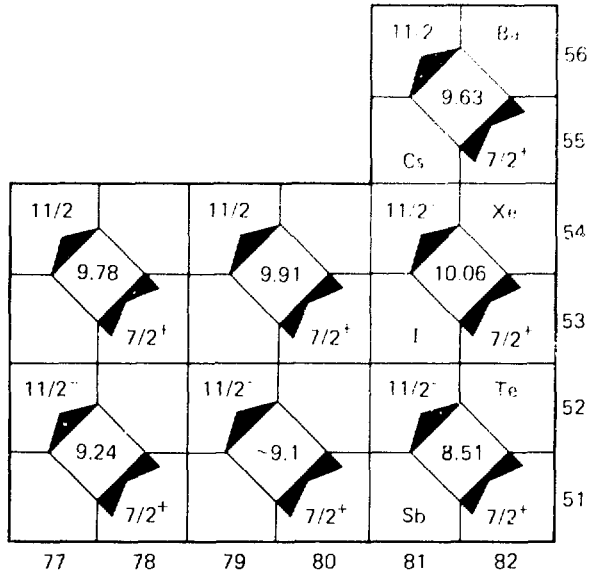
Meyer FIG. 4.3.4



(13/2⁺)	90	1990
(11/2⁺)	19	1974
(13/2⁺)	64	1942
	40	1893
11/2⁺, 9/2⁻	~5	1797
(13/2⁻)	~5	1776
(17/2⁻)	52	1729
19/2⁻		1634 9s

$^{133}_{53}I_{80}$

Meyer FIG. 4.3.5



Meyer - FIG. 4.4.1

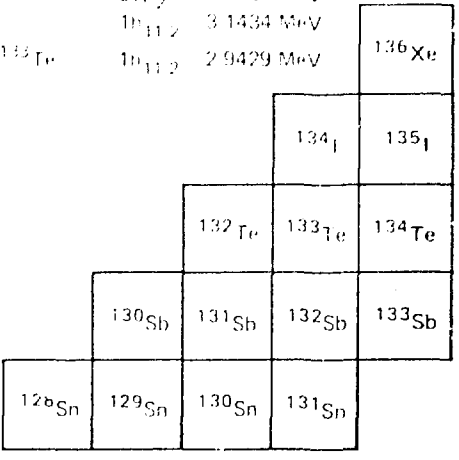
Single particle energies

from a fit of ^{131}Sn

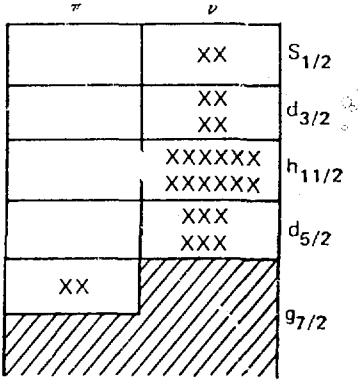
$1g_{7/2}$	4.5030 MeV
$2d_{5/2}$	3.8273 MeV
$2s_{1/2}$	2.1489 MeV
$3s_{1/2}$	3.1434 MeV
$1h_{11/2}$	3.1434 MeV

from a fit of ^{133}Te

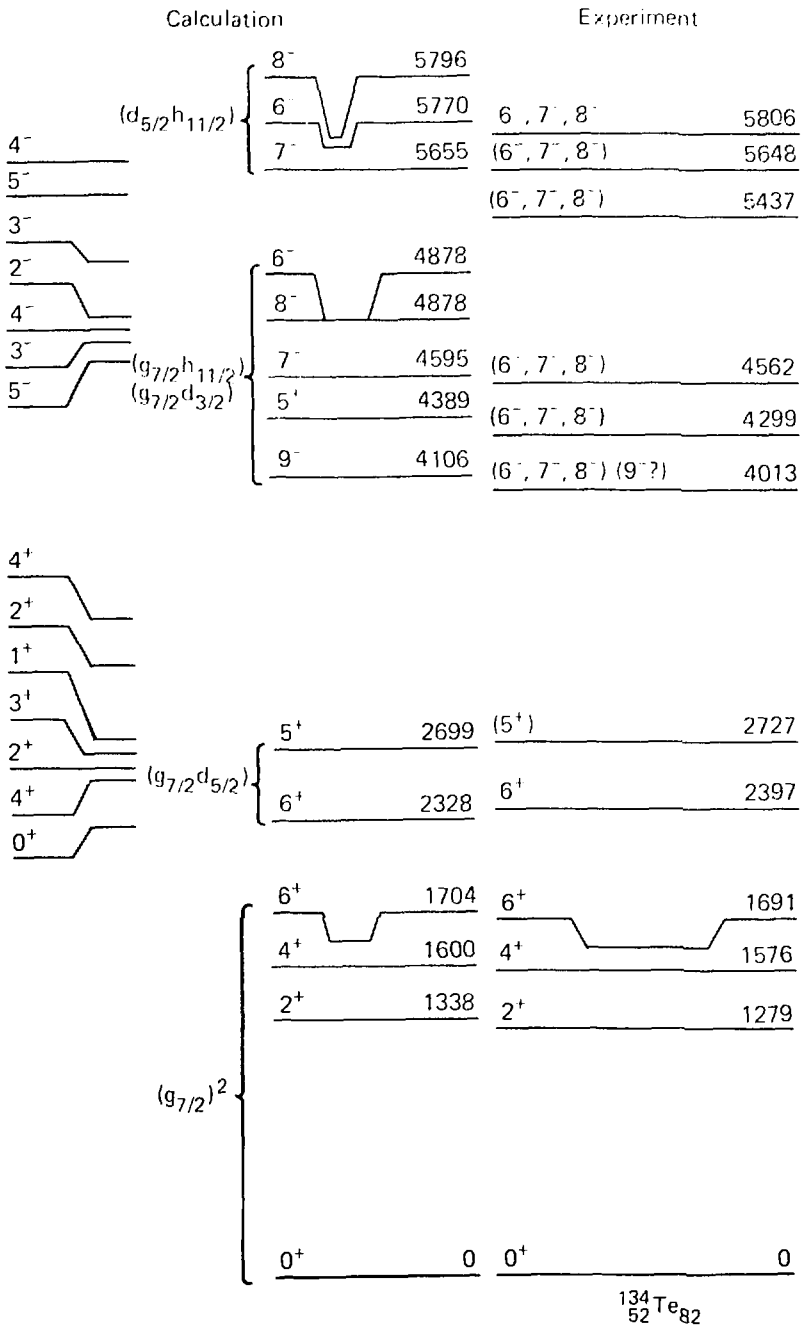
$1h_{11/2}$	2.9429 MeV



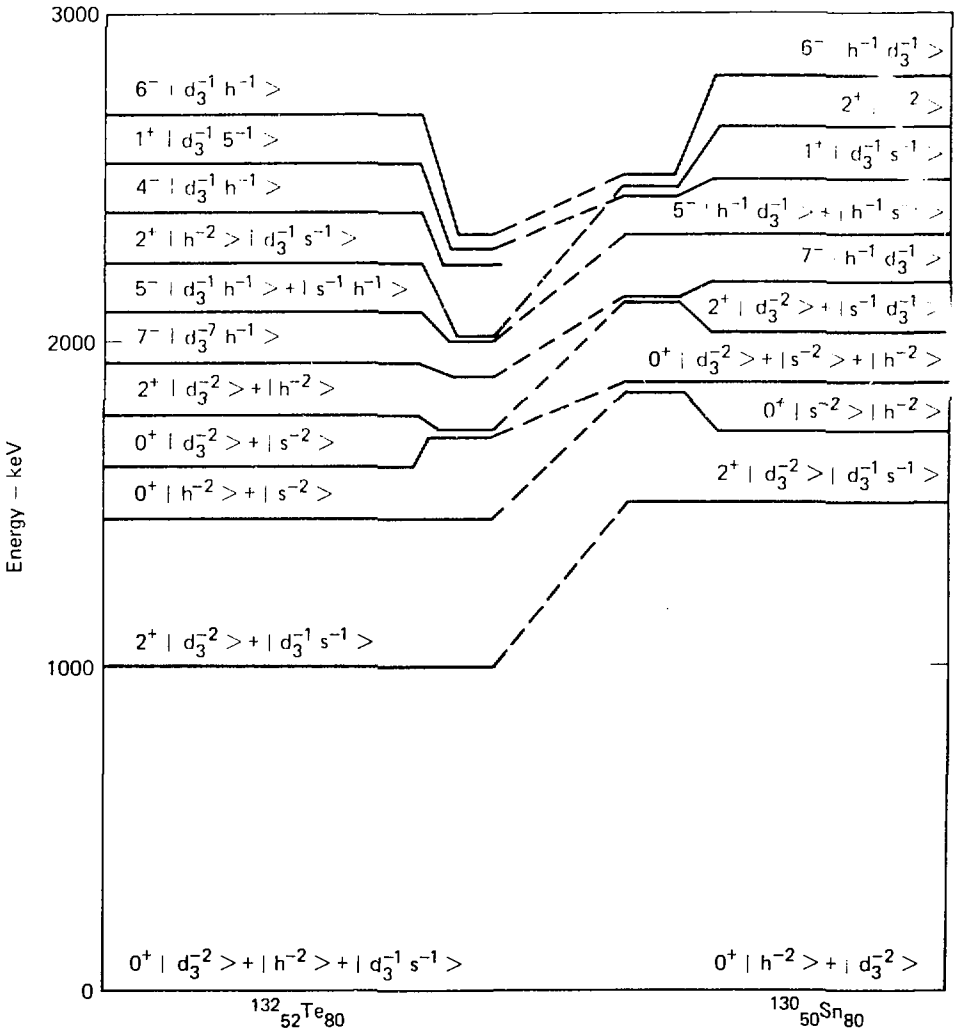
^{134}Te Basis:



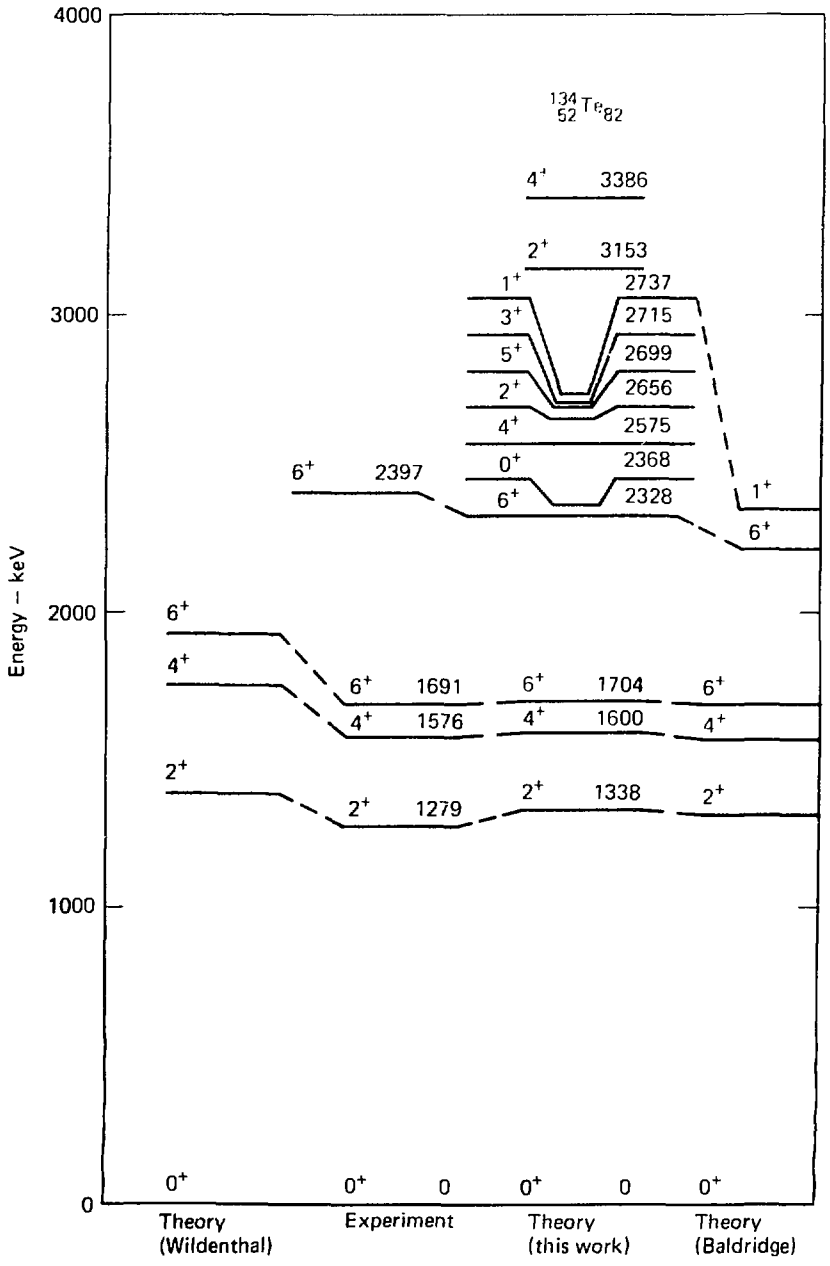
Meyer FIG. A1.1



Meyer FIG. AI.2



Meyer FIG. AI.3



Meyer FIG. AI.4